

PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM JACKFRUIT PEEL

Karthik V. V. S. S



**DEPARTMENT OF CHEMICAL ENGINEERING
NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA**

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of the requirements of the degree of

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in

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by

Karthik V. V. S. S

(Roll Number: 711CH1150)

based on research carried out

under the supervision of

Prof. (Mrs.) Susmita Mishra



DEPARTMENT OF CHEMICAL ENGINEERING
NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA



Department of Chemical Engineering
National Institute of Technology Rourkela

Prof. (Mrs.) Susmita Mishra

May 25, 2016

Supervisor's Certificate

This is to certify that the work presented in the dissertation entitled “*Preparation and characterization of activated carbon prepared from jackfruit peel*” submitted by **Karthik V.V. S. S , Roll Number: 711CH1150**, is a record of original research carried out by him under our supervision and guidance in partial fulfilment of the requirements of the degree of *Master of Technology in Chemical Engineering*. Neither this dissertation nor any part of it has been submitted earlier for any degree or diploma to any institute or university in India or abroad.

Prof. (Mrs.) Susmita Mishra

DEDICATION

Dedicated to the Students, Faculty, and Staff Members of the
Department of Chemical Engineering, NIT Rourkela

DECLARATION OF ORIGINALITY

I, *Karthik V. V. S. S* , Roll Number: 711CH1150, hereby declare that this dissertation entitled “*Preparation and characterization of activated carbon from jackfruit peel*”, presents my original work carried out as a Master of Technology (Dual Degree) student of NIT Rourkela and, to the best of my knowledge, contains no material previously published or written by another person, nor any material presented by me for the award of any degree or diploma of NIT Rourkela or any other institution. Any contribution made to this research by others, with whom I have worked at NIT Rourkela or elsewhere, is explicitly acknowledged in the dissertation. Works of other authors cited in this dissertation have been duly acknowledged under the sections “Reference” or “Bibliography”. I have also submitted my original research records to the scrutiny committee for evaluation of my dissertation.

I am fully aware that in case of any non-compliance detected in future, the Senate of NIT Rourkela may withdraw the degree awarded to me on the basis of the present dissertation.

May 25, 2016

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Karthik. V. V. S. S

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May 25, 2016
NIT Rourkela

Karthik V.V. S. S
Roll Number: 711CH1150

ABSTRACT

Activated carbon in powdered form was prepared from jackfruit peel, a domestic waste. Jackfruit peel having limited economic value has been used as a precursor material to prepare activated carbon. Activated carbon was prepared by the chemical activation of the raw material (Jackfruit peel) which was initially processed and subsequently subjected to activation using varying activating agents such as Potassium Hydroxide (KOH), Zinc Chloride (ZnCl_2) and Phosphoric Acid (H_3PO_4). Surface characteristics such as porosity, iodine number, moisture content, pH, ash content and specific gravity of the prepared activated carbon were estimated. The surface properties from different characterization like FTIR, SEM, TGA analysis indicated that the activated carbon prepared can be utilized as a suitable adsorbent.

Key Words: Activation, Jackfruit Peel, Carbonization, Potassium Hydroxide, Zinc Chloride, Phosphoric Acid

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1.INTRODUCTION

Adsorption is a surface phenomenon by which ions, atoms or molecules of a substance get adhered to the surface of the material, which is known as an adsorbent while the atoms, ions or molecules which get adhered to are known as adsorbates . Absorption involves permeating or dissolving of the absorbent. Adsorption is a surface phenomenon which is of two types :-

Physisorption, where the primary forces involved are Vander Waal's forces. The electronic structure of the atom/ion/molecule does not change on physisorption .

Chemisorption, which is characterized by covalent bonding leads to interaction between the adsorbate and the surface of the adsorbent resulting in the formation of chemical bonds .

A substance that causes substances from gases, fluids, or solids to adhere to its surface without changing the physical properties of the adsorbed substance is called an adsorbent. Activated carbon, silica gel, and activated alumina are some commonly used adsorbents

Activated carbon is amongst the most widely utilized adsorbents today. World demand for activated carbon is very high and the annual production is expected to reach 2.1 million metric tonnes in the next two years. Agricultural waste, for example, rice husk, cassava waste, Nypah palm, palm shells, wood, etc have been accounted for to be suitable for use as precursors in the preparation of activated carbon. In spite of the fact that preparation of activated carbon from these materials has been achieved successfully, yet because of its numerous and widespread applications the interest for activated carbon has increased enormously. Being an extremely efficient and commonly used adsorbent, there is further scope for its preparation from various other

new sources, thereby also leading to the proper utilization of agricultural wastes

One of the drawbacks of activated carbon is that the commercial manufacturing process is not very cost effective. This has led to the search for alternative raw materials, specifically agricultural wastes such as rice husk, groundnut shells, coconut shells, bale fruit shells, jackfruit peel, etc which are not only cheap, but are also environment friendly, for use as precursors in the preparation of activated carbon

Jackfruit is a grown in the tropics and has a wide variety of applications ranging from its extensive use in the preparation of foods like jams, pickles, etc apart from its consumption of its flesh as a fruit. It is also used extensively in the food processing industry. Almost all parts of the fruit, barring its outer peel, are used for various purposes. The peel, which constitutes almost 40 % by weight of an average jackfruit, devoid of any use is often discarded. This peel could be used as a precursor for the preparation of activated carbon.

ORGANIZATION OF THESIS

- The chapter 2 that followed is of Literature Review. It consists of the detailed description of all the concepts related to activated carbon such as adsorption, adsorbents etc. It also consists of citations from various literature sources on topics related to activated carbon.
- Materials and Methods is chapter 3. It consists of the method followed for the preparation of precursor, the process of activation that followed by using different activating agents. This was followed by the method for carbonization at high temperatures. It concludes with the different characterization tests that were performed on the activated carbon samples such as SEM, FTIR, Iodine Number, Carbon Yield etc.
- Chapter 4 is Results and Discussions. It consists of the values of the temperature ranges as deduced from the TGA analysis, followed by the results of various other characteristics like the SEM

analysis, the FTIR analysis, the Iodine number and carbon yield, pH, porosity, etc. and the related discussions.

- The chapter 5 is Conclusion. Various conclusions that could be confirmed from the Results and Discussions section such as the samples with the highest Iodine Number, highest carbon yield and the conditions at which these were achieved were discussed.
- Chapter 6 consists of the Future Scope of Work-the various ways to prepare a better quality product by further optimizing the conditions, the properties of the prepared carbon which could be compared to the already existing samples, the use of activated carbon prepared, which could be used for treatment of various pollutants were discussed.
- Future Scope of Work is followed by the References part, wherein the different literary sources cited in this text were mentioned.

OBJECTIVE

Phosphoric Acid (H_3PO_4), Potassium Hydroxide (KOH), Zinc Chloride (ZnCl_2) are to be used in this study to evaluate the effect of activating agent on the quality and characteristics of activated carbon. The effect of activating temperatures and quantity of activating agent used are also to be evaluated. The significant feature of preparation of activated carbon from Jackfruit Peel which makes it an economical adsorbent is that it can be produced from the waste discarded part i.e the peel, which has limited economic value.

2. LITERATURE REVIEW

2.1 ADSORPTION

Adsorption is caused due to surface energy imbalances. An atom/ion/molecule present in the bulk experiences inter-molecular forces from all directions, whereas an ion/atom/molecule on the surface doesn't experience equal pull from all directions, which result in attracting adsorbates to fulfil its bond requirements.

Dabrowski et al studied the phenomenon of adsorption on phenol and its derivatives with activated carbon being the adsorbent. The extent of adsorption depended on various factors like pore size distribution, type of pores (micropores, mesopores or macropores) , specific surface area, pH of solution and heterogeneity of the phenolic compounds present. [1]

Hongxia et al studied the adsorption behaviour of Th (IV) onto illite. The dependence of adsorption on various factors like contact time, pH, temperature, ionic strength etc was studied. It was found that adsorption in this case, strongly depended on factors like contact time, pH, temperature but independent of the concentration of Humic acid used. [2]

Bhadra et al studied the adsorption of diclofenac sodium from aqueous solutions using oxidized activated carbon. It was found that oxidized activated carbon showed better adsorption capacities than normal activated carbon which could be due to the presence of acidic surface functional groups which are created by oxidation of activated carbon on its surface. [3]

Activated carbon is known to be an extremely useful adsorbent for industrial activities. Activating agents greatly influence the surface

area and porosity of the activated carbon produced. The activating agents normally utilized are H_3PO_4 , KOH, ZnCl_2 , CaCl_2 , NaOH, etc. [4]

2.2 ADSORBENTS

Adsorbents are substances which cause substances to adhere to their surfaces due to energy imbalances experienced by the surface particles of the adsorbent. The particles which get adhered to the surface of an adsorbent are called adsorbates. A good adsorbent should have a high abrasion resistance, high thermal resistance and pore diameters which are small resulting in an adsorbent having a large surface and hence a large adsorption capacity.

According to Torregrosa-Macia et al., a solid adsorbent has to be chemically activated to achieve a relatively wide pore size distribution which results in large adsorption capacities. Physical Adsorption can be used to further enhance the adsorptive capacity of the substance as it involves the process of partial oxidation. [5]

Buekens et al prepared various adsorbents such as activated carbon, silica gel, zeolites, activated alumina, etc and studied the adsorption processes for the removal of pollutants from water bodies and air using these adsorbents. It was found that activated carbon is quite easy to manufacture but it is difficult to adapt it to specific conditions. [6]

Bhatnagar et al studied the suitability of activated carbon and other adsorbents on large scale for the treatment of wastewater. [7]

Although environment friendly and economic adsorbents were developed for treating water pollution, large scale production and widespread use of these adsorbents is yet to take place.

Menéndez-Díaz et al studied the types of carbons and their structures and prepared activated carbon, which is one of the most important

adsorbents, and also studied the physical and chemical properties of the activated carbon prepared. [8]

2.3 ACTIVATED CARBON

Activated Carbon is a highly porous and an amorphous solid consisting of microcrystalline structure with a graphite lattice, usually prepared in small pellets or a powder. A major disadvantage that comes along with the usage of activated carbon is that it gets oxidized at moderate temperatures (over 300°C).

Activated carbon can be prepared from carbonaceous materials, the various sources of which include coal (bituminous, sub-bituminous, and lignite), peat, wood, coconut shells, rice husk, ground nut seed, jackfruit peel etc.

2.3.1 PREPARATION OF ACTIVATED CARBON

Activated carbon can be prepared by physical activation and chemical activation processes. Chemical Activation is preferred over physical activation due to low temperature involved and it being a single step operation.

Abechi et al prepared activated carbon from an agricultural source i.e palm kernel by the method of chemical activation .They were then characterized by standard methods which included SEM analysis and FTIR analysis. [9]

Hameed et al prepared activated carbon using agricultural wastes as precursors. The use of these adsorbents prepared from agricultural wastes finds widespread use as adsorbents in the removal of cationic dyes in textile industries which release solutions containing dyes which are harmful for the environment and aquatic life was studied. [10]

N Bagheri et al prepared activated carbon using corn cob as a precursor. By varying the different parameters, the effect on the

quality of the product was studied. The Iodine Number, Carbon Yield and Specific Surface Area (using BET) of the activated carbon samples prepared were calculated. [11]

Raffiea Baseri et al prepared and studied activated carbon prepared from Cascabela Thevetia (Thevetia peruviana) which was found to have a high adsorption capacity and a very high surface area. Various activating agents like H_3PO_4 , $ZnCl_2$, KOH , $NaOH$, H_2SO_4 . It was found that the high adsorption capacity of Thevetia peruviana activated carbon was due to high porous structure, chemical nature of the surface. The characteristics of activated carbon like porosity, moisture, pH, conductivity, bulk density, specific gravity, porosity, methylene blue number, iodine number and SEM were investigated. The AC prepared was further used as an adsorbent for removal of dyes from waste water released from textile industries. [12]

Verla et al prepared Ac from fluted pumpkin shells at different temperatures using the same concentration of $NaOH$. The prepared activated carbon was characterized using standard tests. Phosphate removal rates of the activated were also studied. [13]

2.3.2 CLASSIFICATION OF ACTIVATED CARBON

Activated carbon is classified into various types based on the size of the carbon particles present.

1. Powdered Activated Carbon (PAC) : Generally, activated carbon are made in the form of powders or fine granules under 1.0 mm in size with an average diameter between 0.15 and 0.25 mm. Therefore they show a very large surface area to volume area with a very small diffusion distance. Powdered activated carbon is characterized as the activated carbon particles held on a 50-mesh strainer (0.297 mm). PAC is comprised of pulverized or ground carbon particles, 95–100% of which will go through an assigned mesh sieve. According to ASTM particles going through a 80-mesh sieve (0.177 mm) and smaller are classified as PAC. PAC is generally not used in a dedicated vessel,

because of the high head loss that it could lead to. Rather, PAC is for the most part added directly to different procedure units, for example, raw water admissions, rapid mix basins, and clarifiers.

2. Granular Activated Carbon (GAC) : Granular activated carbon has a comparatively bigger particle size as compared to Powdered Activated Carbon (PAC) , therefore it has a relatively smaller total surface area as compared to the latter. GAC is mostly used as an adsorbent for gases and vapours as the diffuse rapidly, thereby the smaller surface area is not a hindrance. Granulated carbons are primarily used for waste water treatment, deodorization and separation of components of flow system and in rapid mix systems.

3. Extruded Activated Carbon: Extruded activated carbon is powdered activated carbon bound together by a binder substance, fused and extruded into a cylinder shaped form with diameters varying from 0.8 to 130 mm. It is primarily used as an adsorbent for gas phase applications as gases have low pressure drop, high mechanical strength and low dust content.

4. Bead Activated Carbon: Bead activated carbon is prepared using petroleum pitch and measures in the range of 0.35 mm to 0.8 mm in diameter. It is characterized by its low pressure drop, low dust content and high mechanical strength, albeit with a grain size which is smaller as compared to Extruded Activated carbon. It is the preferred adsorbent for fluidized bed applications owing to its spherical shape.

5. Impregnated Carbon : Activated carbon impregnated with iodine, silver, cations of various metals like Aluminium, Manganese, Zinc, Iron, Lithium etc is called impregnated carbon. Silver loaded impregnated carbon is used for purification of water used for domestic purposes owing to its antimicrobial and antiseptic properties.

6. Polymer Coated Carbon: Activated carbon is coated with a biocompatible polymer which results in a smooth and a permeable coating. It is used in the process of treatment of a patient's blood called as hemoperfusion.

2.3.3. ACTIVATING AGENTS

Activating agents are compounds which are added to the precursor used for preparing activated carbon. The precursors are often soaked in the activating agents. They help in the formation of pores which are essential for the process of adsorption. Also they act as dehydrating agents which remove the bound moisture from the carbonaceous materials, thereby facilitating the formation of cross links which stabilize the substance by making it less prone to volatile losses or volume contraction.

The precursor is soaked in the activating agent before the carbonization takes place. Commonly used activating agents include KOH, ZnCl_2 , H_3PO_4 , NaOH, $\text{Ca}(\text{OH})_2$, etc.

Activated carbon with micro pores was prepared by using Aegle marmelos fruit shell as the precursor and potassium hydroxide (KOH) as the activating agent. The precursor was soaked in the Potassium Hydroxide solution with varying impregnation ratios (0.5 to 2.5) for a duration of 12 hours, thus creating the necessary pores. The samples were then pyrolyzed at high temperatures in a ceramic boat under a continuous flow of nitrogen. [14]

B Ash et al studied the preparation of activated from raw coconut pith using both H_3PO_4 and NaOH. Both the activated samples were characterized and compared with each other. The activated carbon prepared from H_3PO_4 was found to exhibit better properties at lower temperatures as compared to the alkali impregnated samples. [15]

The impacts of activating agent and its concentration on the pore structure and surface chemistry of activated carbons prepared using olive stone with chemical activation done utilizing phosphoric acid as the activating agent were studied. Mass changes were dependent on the impregnation ratio, carbonization temperature and washing procedures that followed. [16]

Activated carbon was prepared from coconut husk using physicochemical activation method which consisted of potassium hydroxide (KOH). The three variables, CO₂ activation temperature, CO₂ activation time and impregnation ratios were varied and its effects on 2,4,6 Trichlorophenol were studied. [17]

Coal tar pitch was used as the precursor for preparation of activated carbon. The pore structure and surface were analysed. Electrochemical properties of AC were also studied using two electrodes in 6mol/L KOH aqueous electrolytes. The effect of activation temperature and KOH/precursor ratio on the pore structure and electrochemical properties were studied in detail. [18]

Sodeinde prepared activated carbon from coconut husks using Zinc Chloride as the activating agent and the activated carbon prepared was used as catalyst in the reduction hexamine cobalt (III) to hexamine cobalt (II). [19]

Yang Yuan et al prepared activated carbon from Chinese fir saw dust using ZnCl₂ as the activating agent under vacuum. The micropore structure, adsorption capacities etc were studied and compared to AC prepared under atmosphere and it was found that AC prepared under vacuum showed higher surface area and pore volume. This was caused due to the fact that the absence of oxygen reduced the secondary reaction of the organic compounds present. [20]

Activated carbon was prepared by Oliveira et al from agro industrial wastes (coffee husks) using Ferric Chloride as an activating agent. The prepared samples were then compared with two other samples-one of which was activated using Zinc Chloride and the other sample activated using a mixture of both Zinc Chloride and Ferric Chloride. [21]

2.3.4 PROPERTIES OF ACTIVATED CARBON

Various properties of activated carbon characterized such as Iodine number, pH, bulk density, porosity etc. Chief among them are Iodine Number and porosity.

Iodine Number-which is the amount in milligrams of iodine adsorbed by one gram of carbon when the iodine concentration in the residual filtrate is 0.1 N .It is one of the most important property which is used to characterize activated carbon. Other important properties include porosity and bulk density .Commercial activated carbon shows an iodine number of 942,while iodine numbers of other A.C prepared from other sources is shown below.

Table 2.1 : Iodine numbers of activated carbon (A.C) prepared from various sources

Raw Material Source	Iodine Number
Coconut Shell A.C [19]	786
Rice Husk A.C [22]	178
Olive Cake A.C [23]	258
Gelam Wood Bark A.C [24]	415

Porosity or void fraction is a measure of the void spaces in a material, and is a fraction of the volume of voids over the total volume, between 0 and 1, or as a percentage between 0 and 100%.It is given by the formula:-

$$\text{Porosity} = (V_v)/(V_t)$$

Where, V_v -volume of the void.

V_t - total volume of the adsorbent.

Porous materials can generally be classified as:-

- microporous materials - pore diameters of less than 2 nm

- mesoporous materials - pore diameters between 2 nm and 50 nm
- macroporous materials - pore diameters of greater than 50 nm

Liu et al studied the structures of three mesoporous substances (order mesoporous carbons) namely, CMK-3, CMK-5, and Si-CMK-5. The adsorption capacities and the porosity of each of the substance was compared with each other and standard commercial activated carbon. [25]

2.3.5 APPLICATIONS

- It is used in the purification electroplating solutions such as the removal of organic impurities from bright nickel plating solutions.
- It is used in the medical field to treat poisonings and overdoses following oral ingestion. It is used in the treatment of diarrhoea, indigestion etc.
- Activated carbon, in 50% w/w combination with celite, is used in low pressure chromatographic separation as stationary phase for separation of carbohydrates by using ethanol solutions as mobile phase in analytical or preparative protocols.
- It is widely used in treating air and water pollution in field as well as industrial processes such as :
 1. Spill clean up
 2. Groundwater remediation
 3. Drinking water filtration
 4. Air purification.

3. MATERIALS AND METHODS

Preparation of activated carbon from jackfruit peel using Potassium Hydroxide as an activating agent is done by the following steps:-

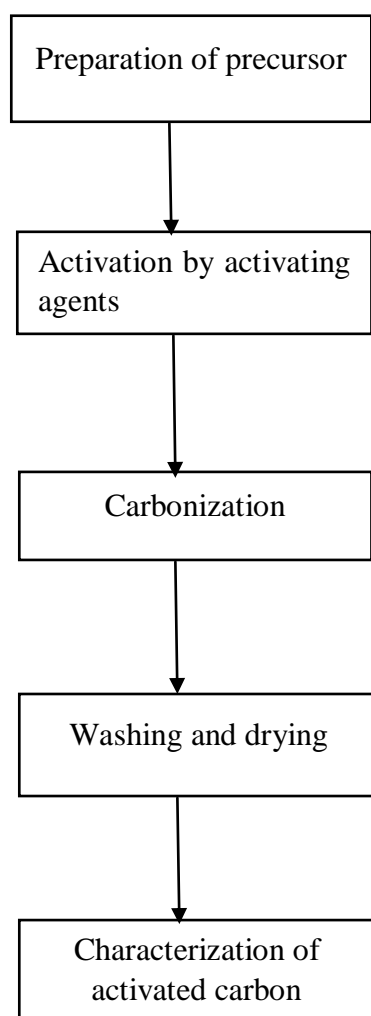


Figure 3.1 : Outline of preparation of activated carbon

3.1 PREPARATION OF PRECURSOR

Small sized jackfruits were collected from the trees on the NIT, Rourkela campus .The outer peel was separated from the inner meat

after which it was cut into small pieces ,washed with water to remove any dust or stains on the peel .This was followed by air drying it for some time and later it was oven dried at a temperature of 70⁰C for 48 hours .Then the peel, devoid of any moisture was crushed using a hammer and then it was ground in a mixer grinder.

3.2 ACTIVATION USING ACTIVATING AGENTS

The grounded samples were impregnated with KOH and ZnCl₂ in different ratios (Weight of impregnating agent/Weight of raw sample) 1:1, 2:1, 1:2. Whereas H₃PO₄ impregnation ratios were taken in terms of percentages-10%, 20%, 30%, 40%, since H₃PO₄ is a liquid. About 10g of impregnating agents were dissolved in distilled water followed by the addition of raw sample in different ratios/percentages accordingly. The samples were left to settle for a period of 12 hours followed by decantation of the samples in which the precipitate was separated from the decant and was transferred to a petri-dish and dried in an oven for a period of 24 hours.

Prior to carbonization certain tests were performed on the samples such as Thermogravimetric Analysis (TGA), Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy

3.3 THERMOGRAVIMETRIC ANALYSIS (TGA)

Activated carbon prepared under different activation condition was subjected to TG analysis to select the temperature range for activation to continue using varying activating agents. Thermogravimetric analysis (TGA) analyses the changes in physical and chemical properties of materials with respect to a function of increasing temperature (at constant heating rate), or as a function of time (at constant temperature and/or constant mass loss). It was used to calculate the temperature ranges between which the carbonization was to be performed.

TGA also provides information on various physical phenomena like vaporization, sublimation, absorption etc. It is used to measure three parameters:- mass change, temperature change, and temperature change. It consists of a precision sample which is to be filled with the sample, and a furnace which is programmable. It can be programmed either to maintain a constant heating rate or a constant mass loss rate with time. Constant heat loss rate is more commonly used.

TG Analyses were performed on the different samples of KOH impregnated precursor (1:1, 2:1, 1:2 samples) and ZnCl_2 samples (1:1, 2:1, 1:2) along with the raw sample and the temperature ranges at which the carbonization process is to be carried out were deduced from the analysis. These temperatures also indicate the temperatures at which there is complete loss in moisture content, lignin, cellulose, and hemi-cellulose.

3.4 CARBONIZATION

The samples impregnated with KOH and ZnCl_2 were kept in a crucible with a lid and heated in their respective temperature ranges as obtained from TGA analysis at every 50°C interval, in a Tubular furnace at heating rate $10^\circ\text{C}/\text{min}$ in an inert atmosphere. The N_2 flow rate for the tubular furnace, which has a maximum temperature of 1400°C , a power supply of 230 Volts, a length of 120 cm, an inner diameter of 50 mm and an outer diameter of 58 mm, is 100 ml/minute and the purity of N_2 is 99.9%. They were kept for a period of 30 minutes at respective temperature. While for H_3PO_4 samples - they were carbonized at a constant temperature of 700°C for a duration of 30 minutes. Later upon finding the Iodine Numbers of the samples, the samples were subjected to carbonization by increasing the duration to 60 minutes and also raising the temperatures of the samples which showed the highest iodine numbers by 50°C .



Figure 3.2: Tubular Furnace used to carbonize the precursor

3.5 WASHING AND DRYING

The samples once removed from the tubular furnace were allowed to cool down to room temperature and subsequently washed with 0.1N HCl till no change in pH of the washed samples were observed. The samples were subsequently dried at a temperature of 100°C to remove the moisture content

3.6 SCANNING ELECTRON MICROSCOPE

A scanning electron microscope (SEM) produces images of a sample by scanning it with a beam of electrons. On interaction, the electrons produce images of the sample that contain information of the composition and surface's topography. SEM has a resolution of better than 1 nm. Specimens can be observed under a wide range of temperature and pressure conditions. SEM produces a wide range of signals such as secondary electrons, characteristic X-Rays, specimen current, back scattered electrons, etc, the most important of them being the secondary electrons. SEM produces a wide range of magnifications ranging from 10 times to over 5,00,000 times which is much higher than the most powerful light microscopes.

3.7 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

Fourier transform infrared spectroscopy obtains an infrared spectrum of absorption or emission of a solid, liquid or gas. It is used to find the functional groups present in the sample.

The aim of absorption spectroscopy is to check how well light at different wavelengths is absorbed by the sample. FTIR was used to shine a beam of light containing many frequencies on the sample and measure the amount of light absorbed by the sample. Next, a beam with different frequencies of light was incident on the samples which gave another data point, and so on.

The beam of light used was generated by using a broadband source of light (one which contains full spectrum of wavelengths of light) which was then focussed on to Michelson Interferometer-which contained mirrors in a certain combination and one of which was moved using a motor. Some of the wavelengths, due to the movement of the mirrors, got periodically blocked, transmitted due to the phenomenon of wave interference. The wavelengths were then modulated, thereby allowing each beam coming out of the interferometer to have a different spectrum of wavelengths.

FTIR was performed on the raw sample, H_3PO_4 impregnated sample, KOH impregnated sample, ZnCl_2 impregnated sample. All the samples were then compared with the raw sample, thereby detecting the different functional groups present in the samples that were activated using the above mentioned activating agents.

3.8 DETERMINATION OF IODINE NUMBER AND CARBON YIELD

Iodine Number :

Iodine number tests were performed on the samples by standard Iodine Number test. It was performed in the following way:

Reagents:

- 0.1N Iodine solution (40 gm KI in 1 Litre of Distilled water).

- 0.05N Sodium Thiosulphate solution (12.5 gm $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in 1 Litre of Distilled water)
- 1% Starch solution
- Activated carbon

Procedure:

I -Standardization of Iodine solution

- 10 ml of 0.1N Iodine solution was taken in a conical flask.
- 2 drops of Starch solution was added to it
- The pale yellow colour of Iodine Solution turned Blue.
- It was titrated against 0.05 N Sodium Thiosulphate till it became colourless.
- Burette reading corresponded to blank reading.(B).

II

- 0.2 gm of Activated carbon was weighed.
- It was introduced into a completely dry volumetric flask.
- 40ml of 0.1N Iodine solution was added
- The flask was shaken continuously for 4 minutes and filtered.
- The filtrate was collected in a dry flask and 10 ml of the filtrate was titrated against Standard Sodium thiosulphate solution using starch as indicator
- Burette reading corresponded to (A).

Calculations:

Iodine value= C x Conversion factor; mg/gm

Conversion Factor: (Molecular weight of iodine (127) x normality of iodine x 40) / (Wt. of carbon x Blank reading)

Where, C=B-A.

Carbon Yield :

A crucible was filled with known amount of precursor impregnated with activating agent. It was subjected to activation and then again the weight of the activated carbon was estimated. The loss in weight with respect to initial weight gave the yield of the activated carbon .

$$X = (\text{Initial weight}-\text{Final weight})/(\text{Initial Weight})$$

$$\text{Carbon Yield \%} = (1-X) \times 100$$

3.9 OTHER CHARACTERISTICS

Other characteristics of the prepared activated carbon such as pH, porosity, moisture content, ash content and volatile matter etc were determined using the standard tests of for each of the above mentioned characteristic

3.9.1 DETERMINATION OF pH:

The pH was determined using the standard test. 100 ml of distilled water was added to 1.0 gram of Jackfruit Peel Activated carbon (JPAC) in a beaker and the mixture was stirred for one hour. Later, upon stabilizing the pH of the samples was measured using a pH meter.

3.9.2 DETERMINATION OF MOISTURE CONTENT:

1g of activated carbon was taken in a petri-dish and kept in an oven at a temperature of 120⁰C for a duration of 4 hours. The loss in weight of the sample gave the moisture content.

$$\text{Moisture Content} = (1-\text{Final weight}) \times 100.$$

3.9.3 DETERMINATION OF ASH CONTENT AND VOLATILE MATTER:

A crucible was pre heated in a muffle furnace to about 700⁰ C, then cooled and weighed. 1.0 g of jackfruit peel activated carbon (JPAC) samples were transferred into the crucibles and re-weighed. The crucibles containing the samples were in a muffled furnace and heated to 700⁰C. It was removed and allowed to cool in a desiccator to room temperature. The ash content was calculated using the given formula:

$$\text{Ash (\%)} = (\text{ash weight}) / (\text{oven dry weight}) \times 100.$$

$$\text{Volatile Matter (\%)} = (100 - ((\text{Carbon Yield \%}) + (\text{Moisture Content \%}) + (\text{Ash \%}))).$$

3.9.4 DETERMINATION OF DENSITY:

A known weight of the activated carbon was taken in a measuring cylinder and its volume was measured.

Density =(mass/volume)

Similarly, specific gravity was calculated by :-

Specific Gravity = (density of activated carbon/density of water).

Density of water = 1000 g/cm³.

3.9.5 DETERMINATION OF POROSITY:

The jackfruit peel activated carbon (JPAC) was put in an oven for one hour at a temperature of 100°C and allowed to dry .A clean, dry, well corked density bottle was weighed and filled with water and reweighed again. Samples of activated carbon were taken and ground to powder and put into the bottles with little water and the weight was measured again.

Porosity = V_v/V_t , where V_v = volume of void, V_t = total volume.

$V_v = V_t - V_s$, where V_s is the volume of solid sample taken.

3.9.6 DETERMINATION OF SPECIFIC SURFACE AREA

Specific surface area is a characteristic property of solids and it is measured as the amount of surface area of a substance per unit mass. The surface area is measured on the basis of BET theory which itself is based on the Langmuir theory. It is measured using by using Autosorb 1 surface analyzer. Nitrogen gas is used as the adsorbate and the process is carried out at 77 K.

4. RESULTS AND DISCUSSIONS

4.1 CHARACTERISTICS OF ACTIVATED CARBON

The various characteristics of the activated carbon prepared from jackfruit peel such as Thermogravimetric analysis (TGA) which determines the temperature required for carbonization, Scanning Electron Microscopy (SEM) which gives the images of the porous surface of the activated carbon prepared, Fourier Transform Infrared Spectroscopy (FTIR) –which helps in determining the functional groups present in the activated carbon sample. Other important characteristics such as iodine number, carbon yield, moisture content, ash content and volatile matter, density, porosity etc were also determined

4.1.1 THERMOGRAVIMETRIC ANALYSIS

Thermogravimetric Analysis graphs of KOH impregnated samples in different ratios (KOH:precursor)

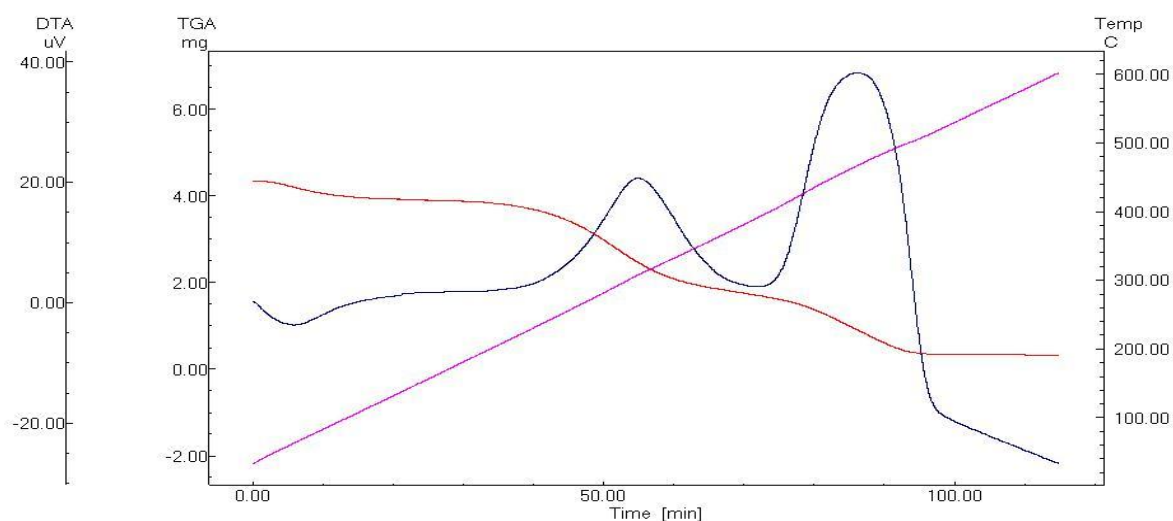


Figure 4.1 (a): TGA graph of raw sample

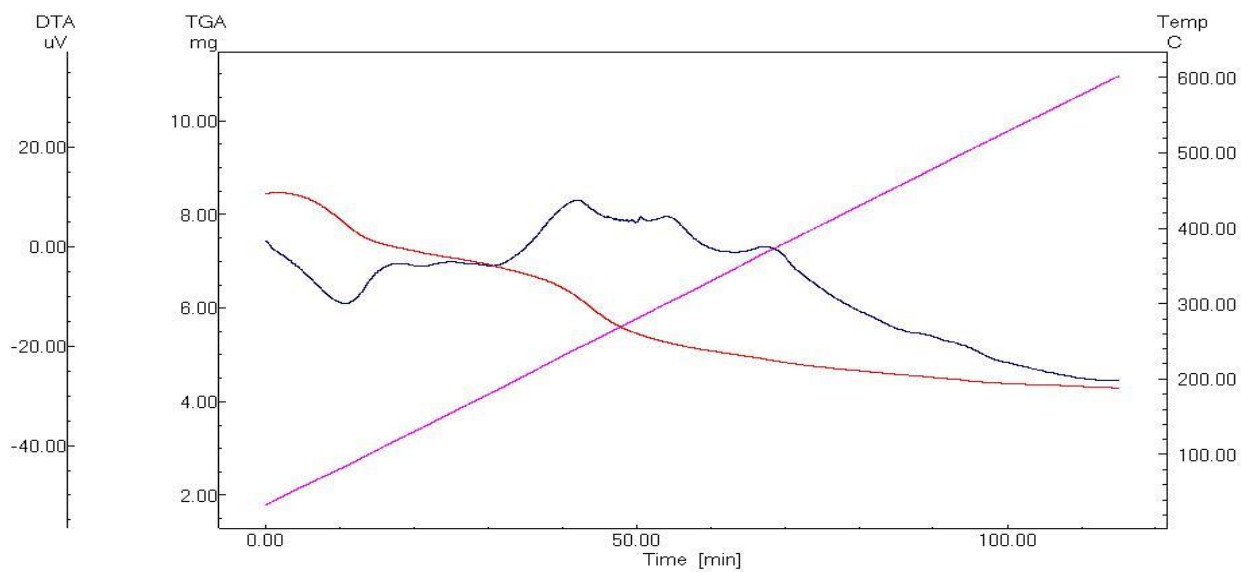


Figure 4.1 (b) : TGA graph of 1:1(KOH : precursor) impregnated sample

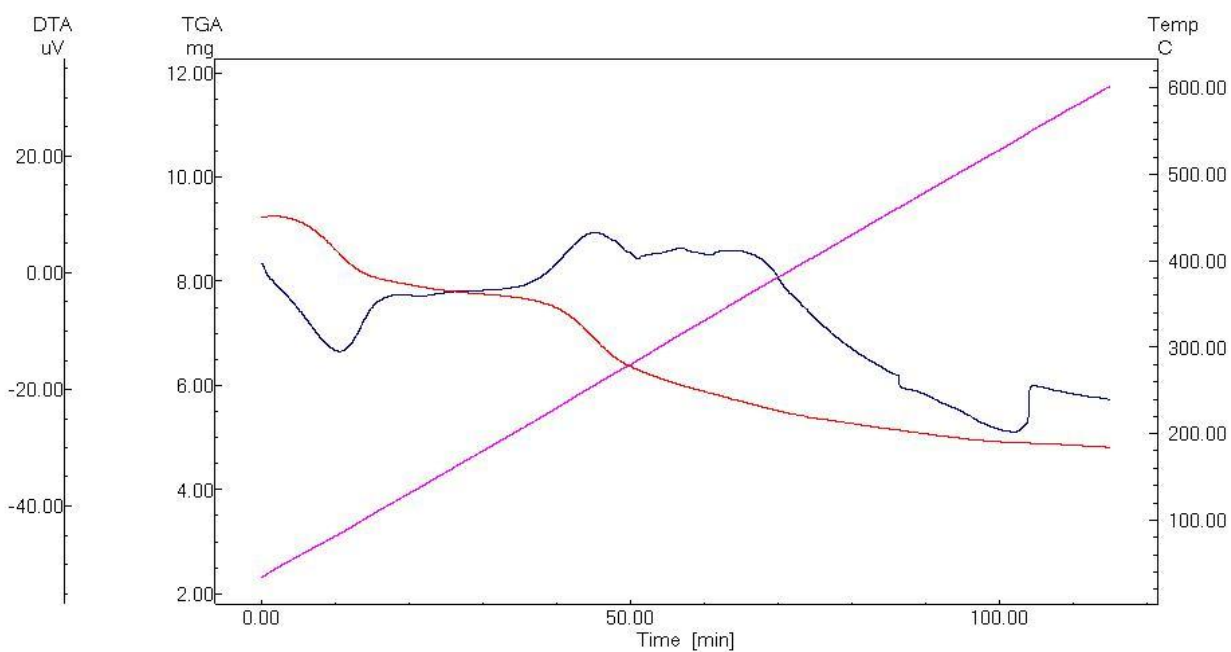


Figure 4.1 (c): TGA graph of 2:1(KOH : precursor) impregnated sample

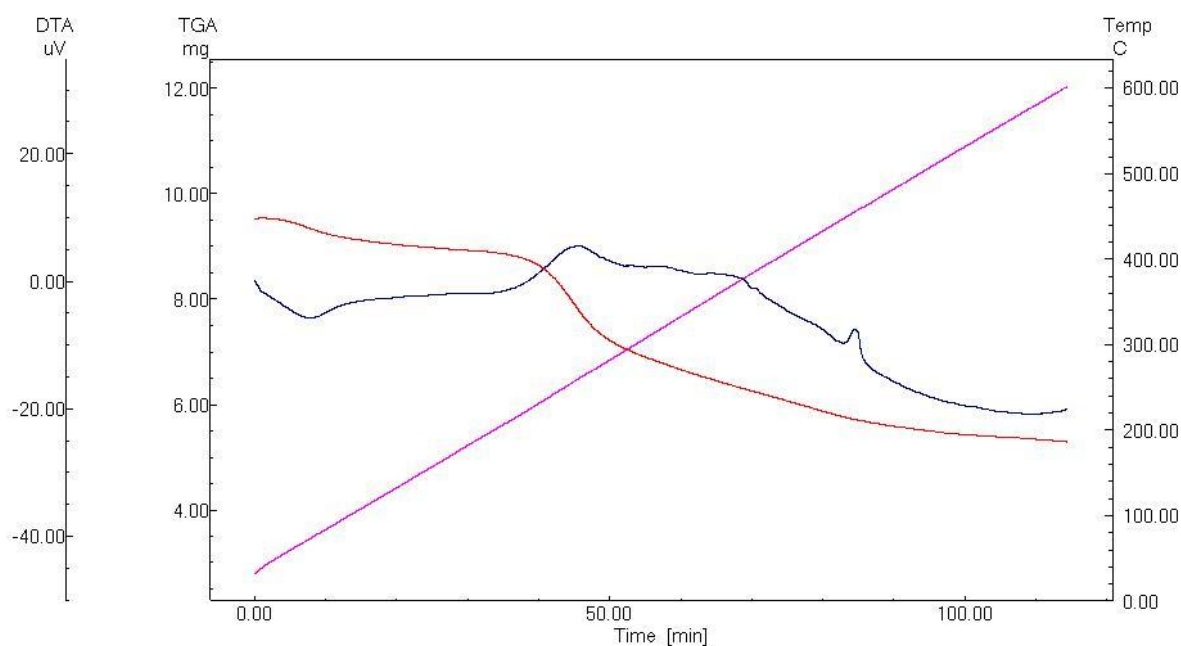


Figure 4.1 (d) : TGA graph of 1:2 (KOH :precursor) impregnated sample

The temperature ranges for each of the samples impregnated with KOH as the activating agent were deduced from the graphs and carbonization was carried out accordingly. Table-2 below provides the temperature range obtained from TGA for different impregnation ratio with KOH as impregnating agent.

Table 4.1 : Effective temperature range at different IRs of KOH for carbonization

Impregnating Ratios (IR)	Potassium Hydroxide
1:1	250°C-500°C
2:1	250°C-450°C

1:2	250°C-400°C
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Thermogravimetric Analysis graphs of ZnCl_2 impregnated samples in different ratios (ZnCl_2 :precursor)

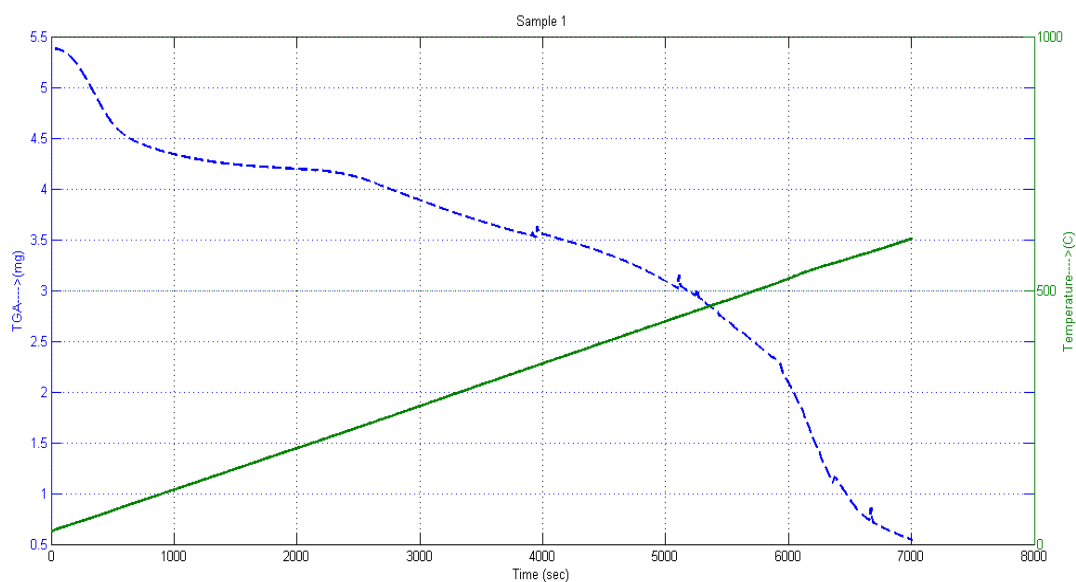


Figure 4.2 (a) : TGA graph of 1:1(ZnCl_2 :precursor) impregnated sample.

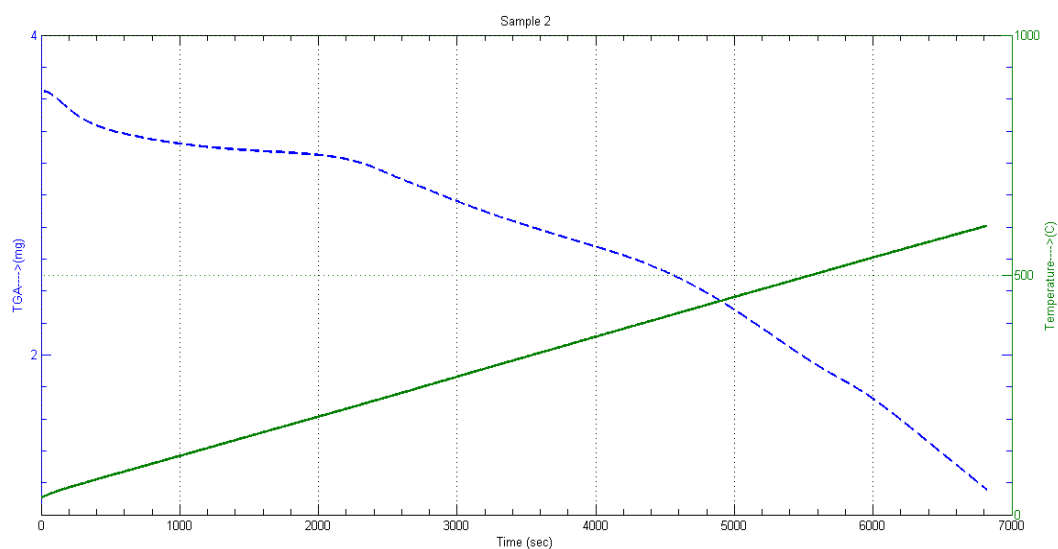


Figure 4.2 (b): TGA graph of 2:1(ZnCl_2 :precursor) impregnated sample

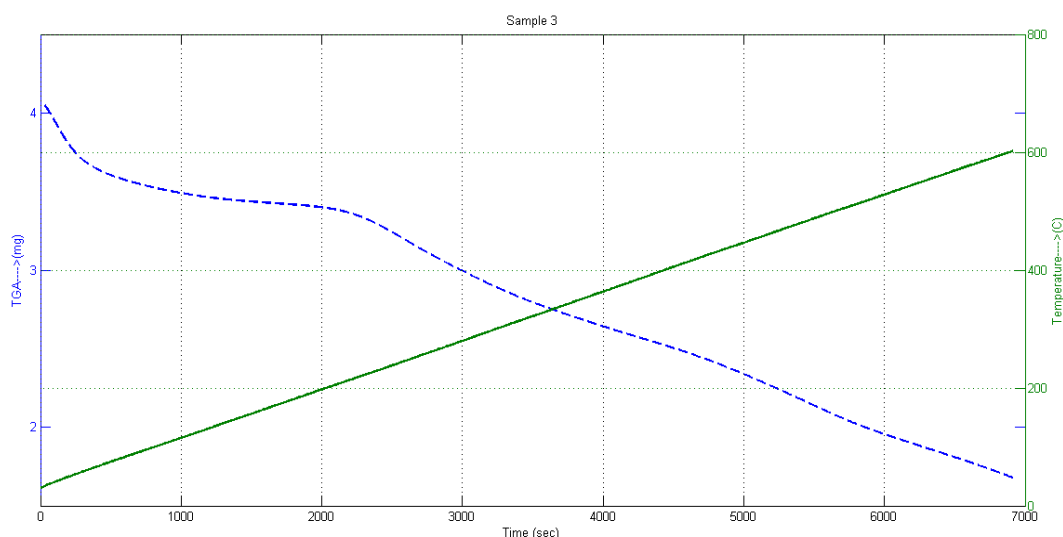


Figure 4.2 (c): TGA graph of 1:2 (ZnCl_2 :precursor) impregnated sample

Table 4.2 : Effective temperature range at different IRs of ZnCl_2 for carbonization

Impregnation Ratio	Temperature Range
1:1	500°C-650°C
2:1	500°C-650°C
1:2	400°C-600°C

Carbonization was carried out for different samples according to the temperature ranges in the tables above. The moisture, lignin, cellulose and hemi-cellulose contents in the samples are completely removed thus making it conducive for carbonization in these temperature ranges.

4.1.2 SCANNING ELECTRON MICROSCOPY

The porous structure of the carbonized samples was studied using the Scanning Electron Microscope. Below are the images of the samples

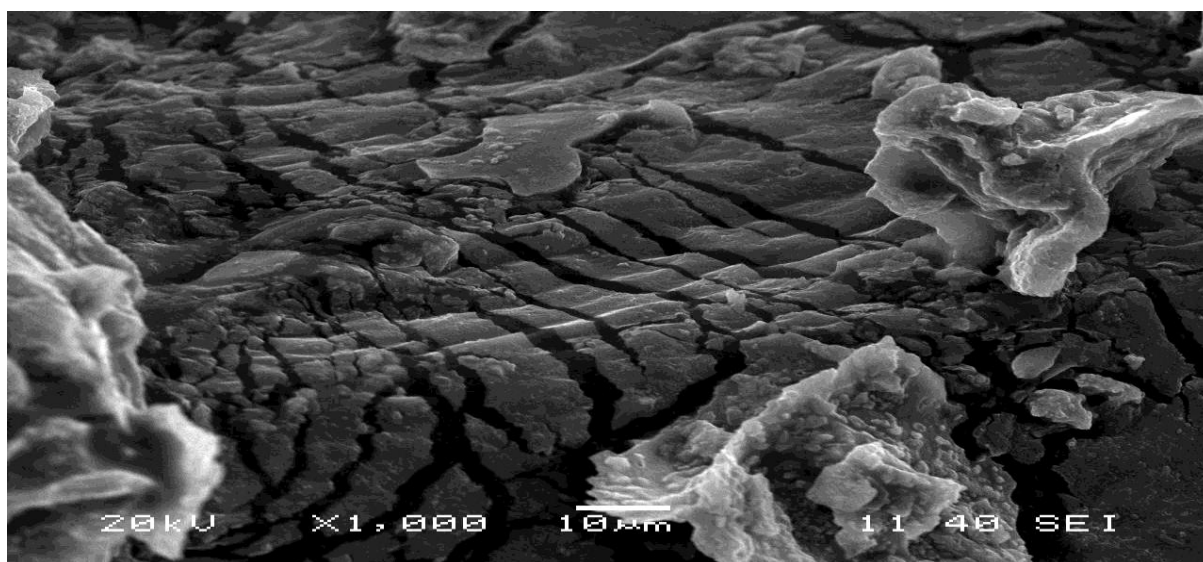


Figure 4.3 (a): SEM image of raw sample

The image of the raw sample above shows no pores. An activating agent is required to help the formation of pores. The bound moisture present could be removed by the use of activating agents like H_3PO_4 , KOH , ZnCl_2 etc.

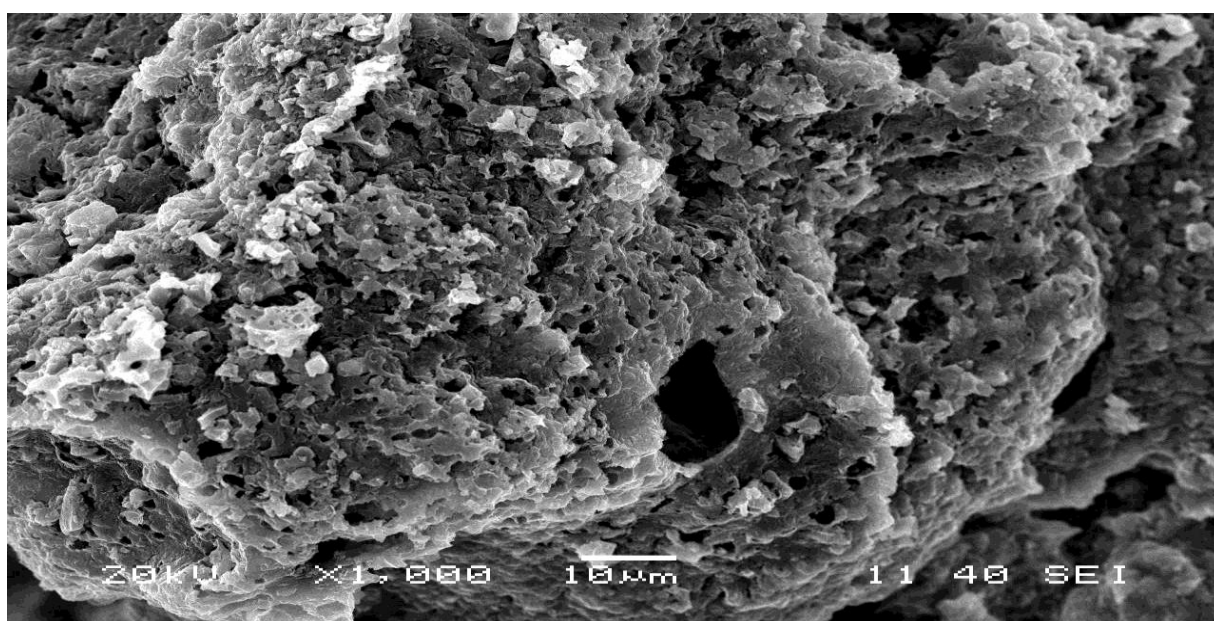


Figure 4.3 (b): SEM image of 1:1(KOH :precursor) impregnated sample at 500°C

Above is the SEM image of the KOH impregnated activated carbon in the ratio 1:1 at a temperature of 500°C. A few pores can be seen in the diagram. The micropores are small in size, which owing to the high

temperatures and the presence of activating could further expand to form larger mesopores. The addition of activating agent and high temperature could further help in the formation of more micropores which could be a result of removal of traces of bound moisture and other volatile components and the biopolymers present in the raw material.

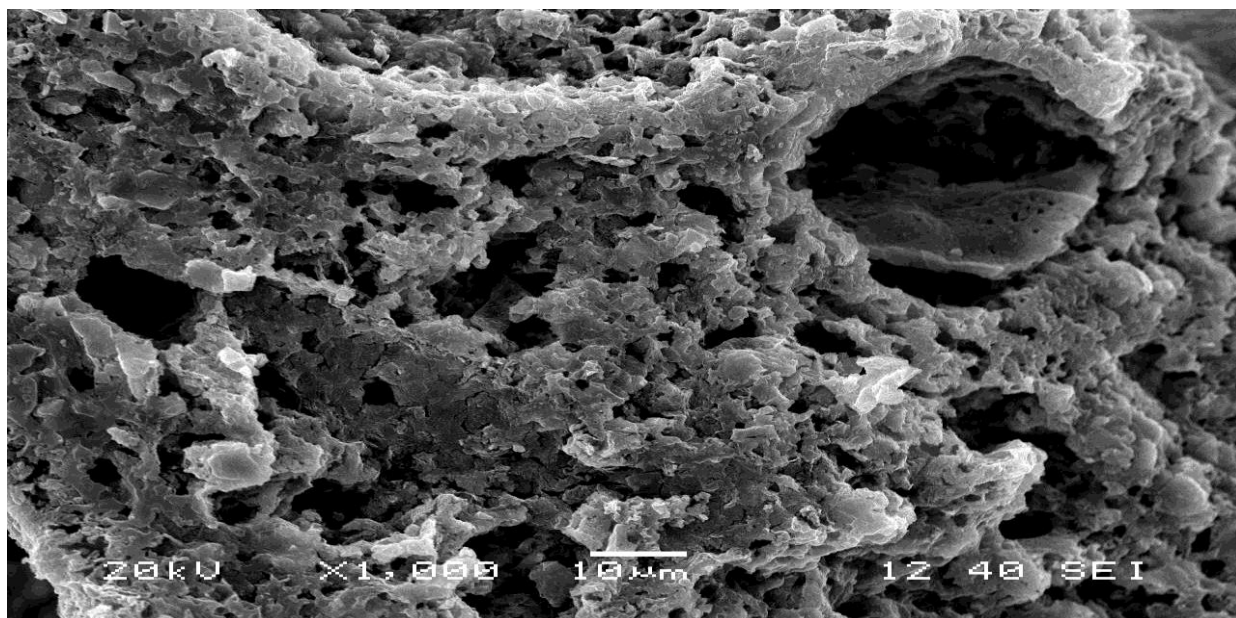


Figure 4.3 (c): SEM image of 2:1(KOH :precursor) impregnated sample at 450°C

The above image is that of activated carbon prepared using KOH as an activating agent with an impregnation ratio (KOH :raw sample) of 2:1. It can be seen that with the increase in the pores could be attributed to a number of reasons. Firstly the high temperature of 450°C, could have resulted in the escaping of the bound moisture and other volatile matter from the precursor material leaving voids. Secondly, the large macromolecules of the precursor material breakdown and reconstitute (sometimes forming aromatic compounds at higher temperatures) which could have led to the formation of pores. Another important factor is that of the activating agent present (KOH) which could have reacted with carbon skeleton of the precursor resulting in the formation of pores and enlargement of the existing ones.

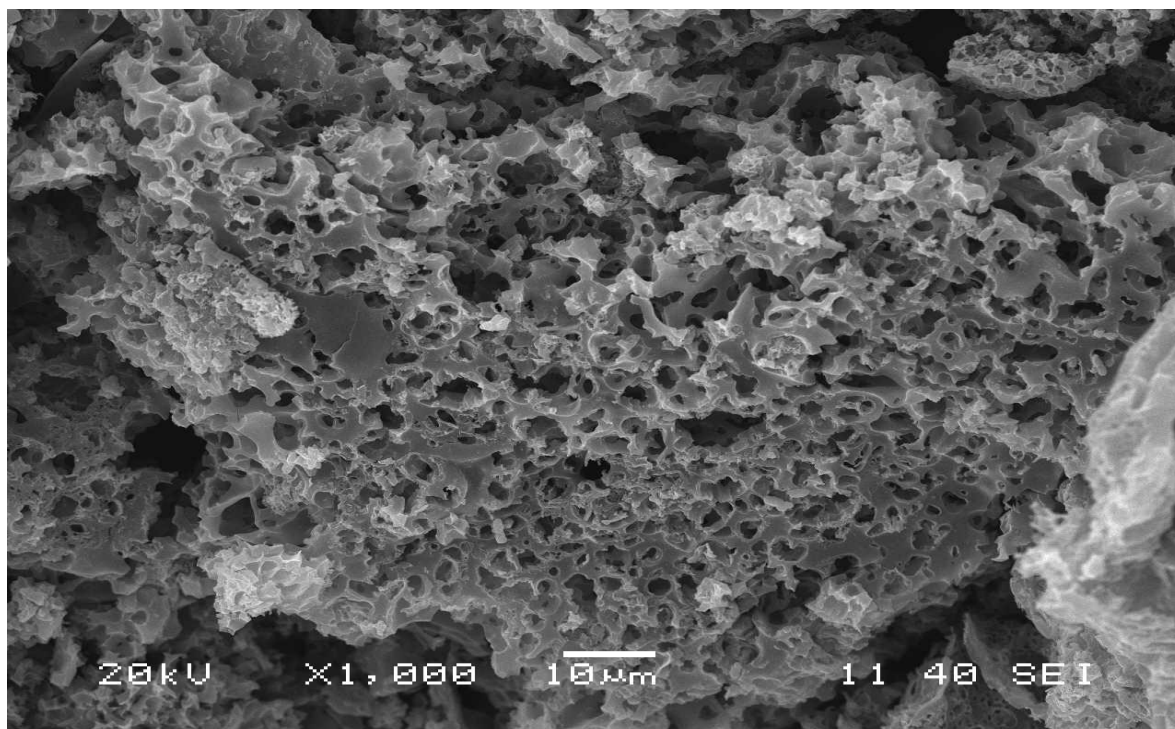


Figure 4.3 (d) : SEM image of 1:2 (KOH :precursor) impregnated sample at 500°C

In the above image of activated carbon prepared using KOH as the activating agent in a ratio of 1:2 at a temperature of 400°C for a duration of 30 minutes, it is observed that it has comparatively lesser number and less pronounced pores are formed which is the result of use of KOH in lesser quantity as compared to the previous sample. Also owing to the slightly lower temperature in this case, lesser number of mesopores can be observed.

SEM images of ZnCl_2 impregnated samples in different ratios

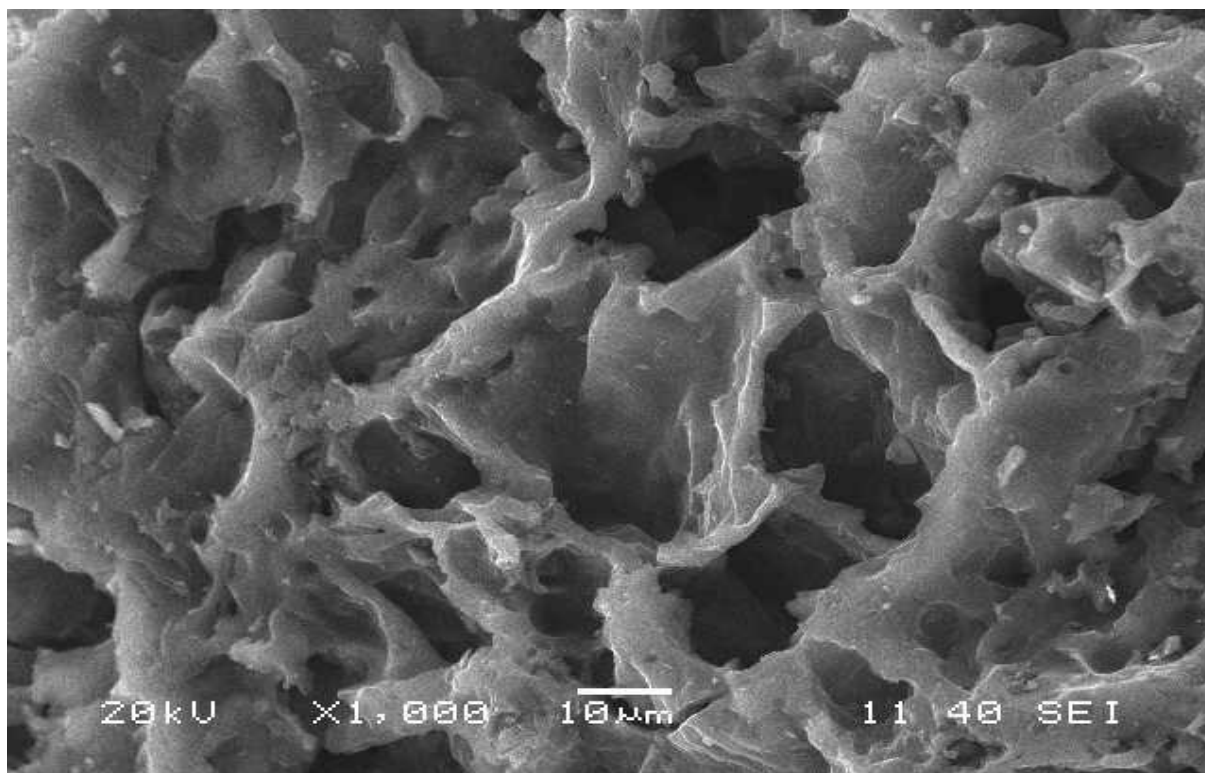


Figure 4.4 (a) : SEM image of 1:1(ZnCl_2 :precursor) impregnated sample at 650°C

The above image is that of activated carbon prepared by using jackfruit peel as a precursor and ZnCl_2 as the activating agent with an impregnating ratio of 1:1 (ZnCl_2 :precursor). It is noticed that the pores are larger in size as compared to the KOH owing to the higher temperatures. Also the pores are found to be less pronounced due to the fact that Zn present in ZnCl_2 is not an alkali metal. The alkali cation due to their highly reactive nature leads to the formation of larger number and more pronounced pores.

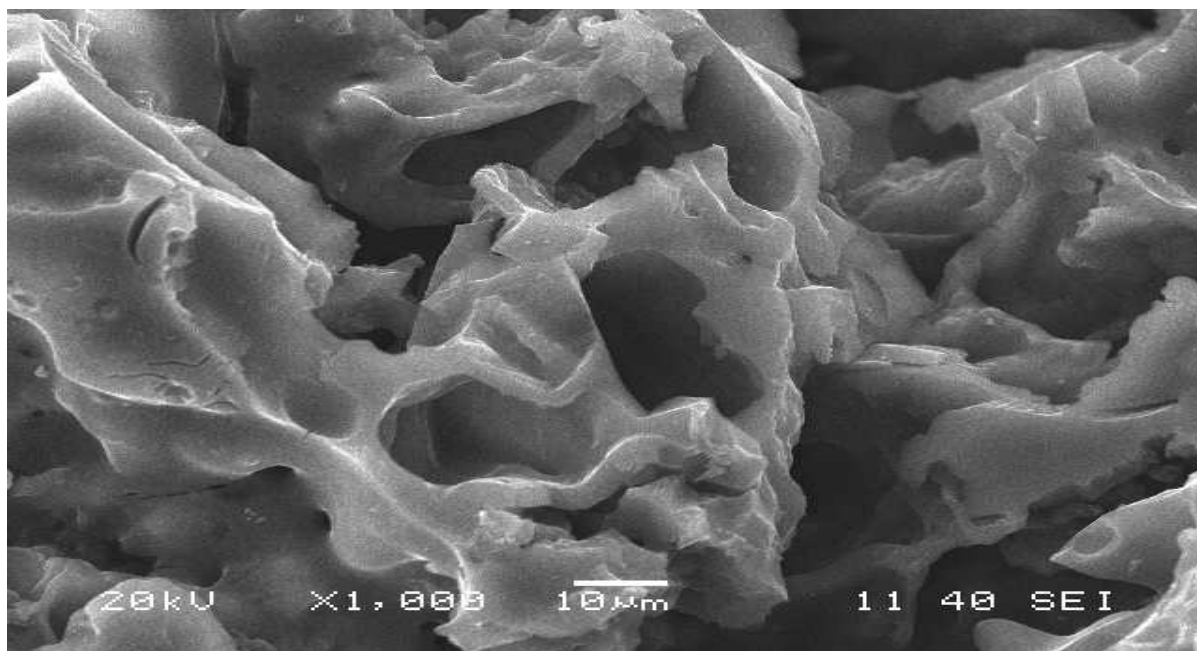


Figure 4.4 (b) : SEM image of 2:1(ZnCl_2 :precursor) impregnated sample at 650°C

Although this sample is carbonized at the same temperature as the previous sample, the presence of ZnCl_2 in larger quantities (2:1 ratio) results in formation of pores of larger size, which affects the adsorptive power.

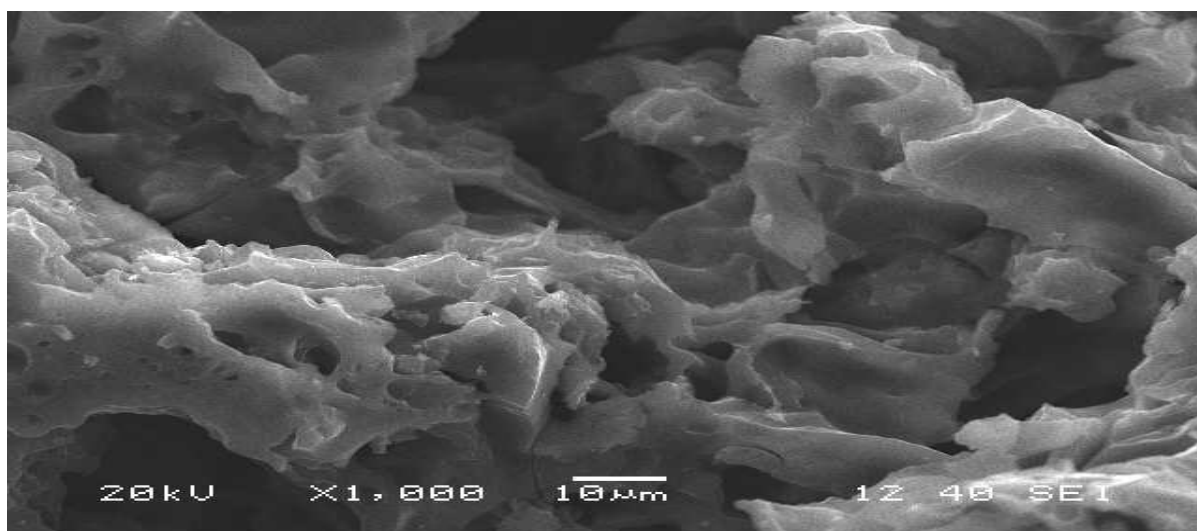


Figure 4.4 (c): SEM image of 1:2 (ZnCl_2 :precursor) impregnated sample at 600°C

The above image is that of activated carbon prepared by using ZnCl_2 as the impregnating agent at a temperature of 600°C . It is observed that the

thermal stresses caused due to the high temperatures result in the formation of cracks and crevices which ultimately lead to the formation of pores. The lower temperature (600°C) as compared to the previous samples and the lesser quantity of ZnCl_2 used resulted in the formation of lesser number of pores.

Overall it is observed that the KOH impregnated samples which are carbonized at lower temperatures develop smaller micropores in larger quantities. Here KOH is the more dominating factor as compared to temperatures. While in case of ZnCl_2 , the samples developed larger sized pores which is the result of high temperatures (temperatures approaching 700°C) at which more macromolecules breakdown into aromatic ring compounds thus leaving larger vacant lots, leading to the formation of larger pores.

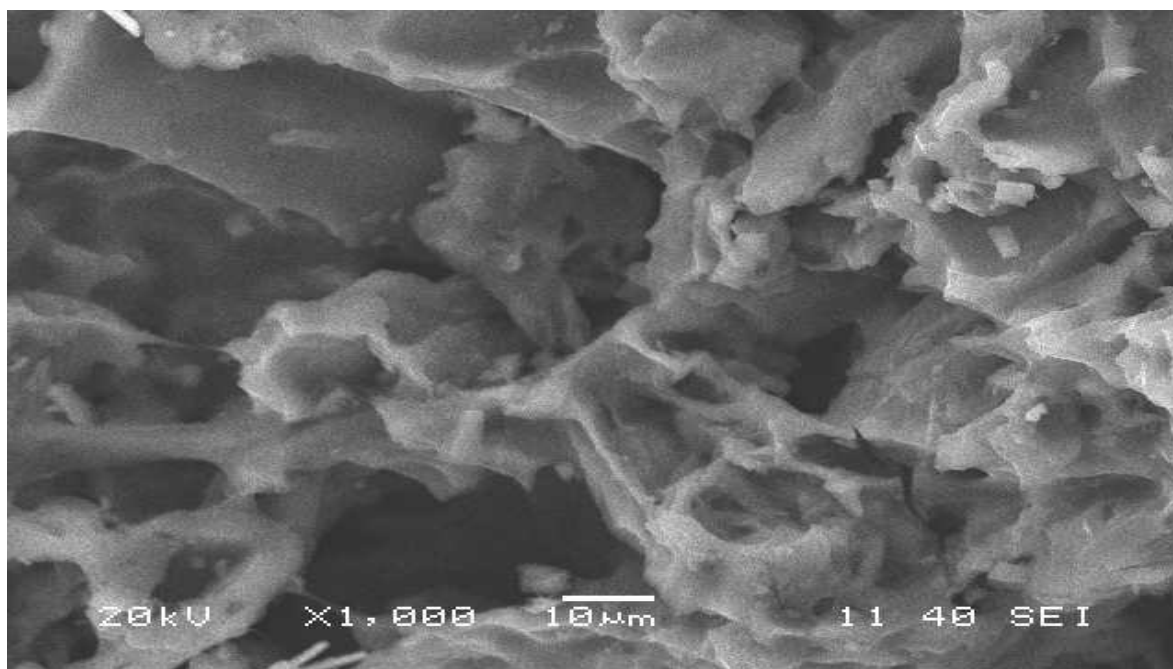


Figure 4.5 : SEM image of 30% H_3PO_4 activated carbon at 700°C

The above image of H_3PO_4 activated carbon at 700°C shows pores of larger size as due to the high temperatures the smaller pores could have merged into larger ones and the macromolecules were further broken down leading to the formation of aromatic compound which resulted in the large pores being formed.

4.1.3 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

FTIR analysis is performed to find the functional groups of carbon present in the samples. The functional groups of the wavelengths at which prominent crests and troughs are formed were found. Each of the samples (H_3PO_4 , KOH, ZnCl_2 impregnated samples) were compared with the raw sample.

FTIR graphs of the samples in comparison with the raw sample

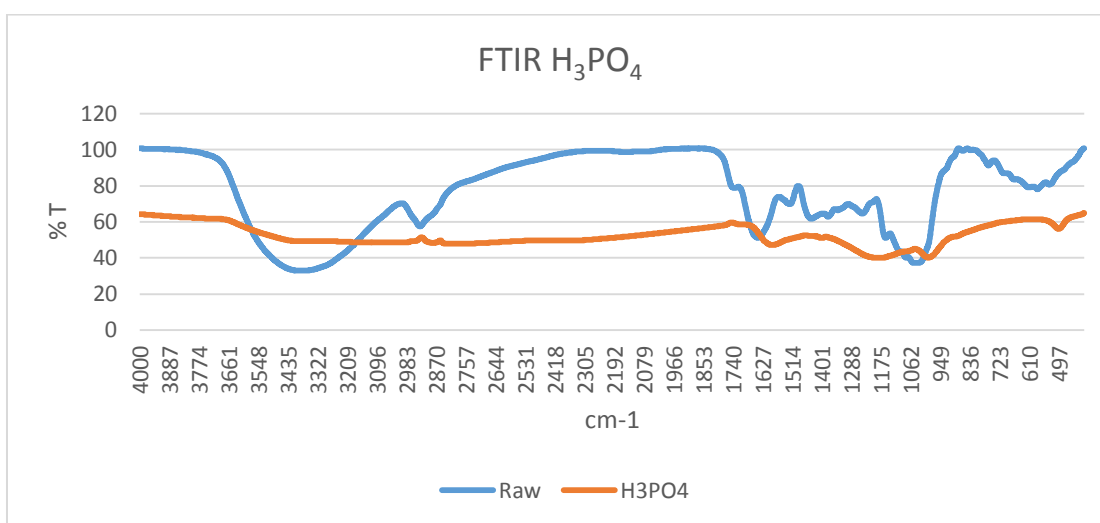


Figure 4.6 (a): FTIR graph of H_3PO_4 sample

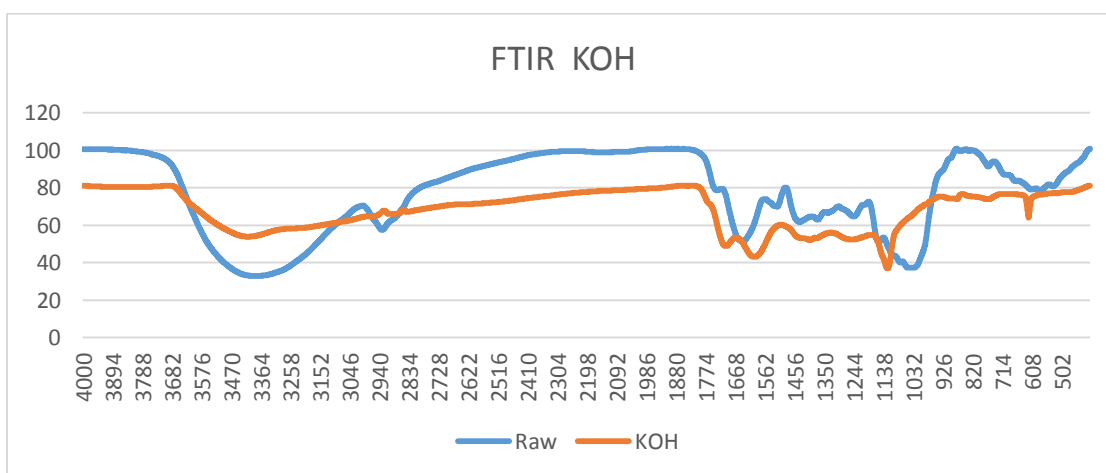


Figure 4.6 (b): FTIR graph of KOH sample

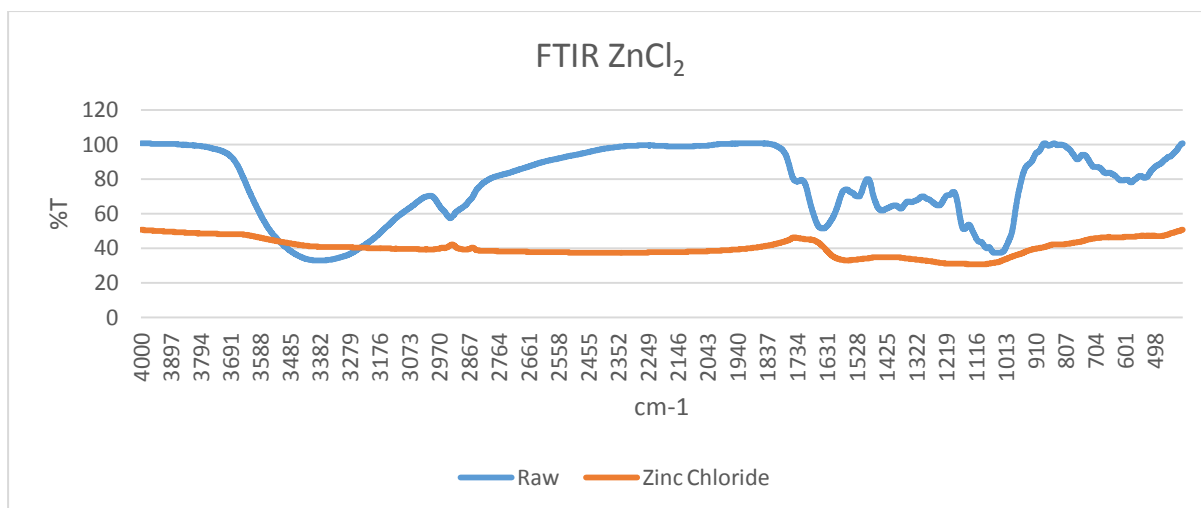


Figure 4.6 (c): FTIR graph of ZnCl₂ sample

Table 4.3 : List of Functional groups present in each sample from FTIR Analysis

Raw		H ₃ PO ₄		KOH		ZnCl ₂	
cm ⁻¹	Functional Group	cm ⁻¹	Functional Group	cm ⁻¹	Functional Group	cm ⁻¹	Functional Group
498	C-I	498	C-I	601	C-Cl	807	Alkene
910	Alkene	910	Alkene	1116	C-O	1013	C-F
1219	C-F	-----	-----	1528	Alkene	1734	Carbonyl
1631	Alkene	1631	Alkene	1734	Carbonyl	2970	Alkane
2867	Alkane	-----	-----	-----	-----	-----	-----
3486	Alcohol	-----	-----	-----	-----	-----	-----

Listed above in the table are the different functional groups present in the samples at each of the major crests and troughs in the graphs.

Below is the FTIR graph of ZnCl₂

4.1.4 IODINE NUMBER AND CARBON YIELD.

Iodine numbers and Carbon Yields of samples impregnated with different activating agents are as follows.

Table 4.4 :Iodine Numbers and Yield of Activated carbon prepared using H_3PO_4

H_3PO_4 (%)	Iodine Number	Carbon Yield (%)
10	860	45.3
20	1005	56.4
30	1235	82.3
40	1220	76.3

Table 5 :Iodine numbers and carbon yield of H_3PO_4 impregnated samples

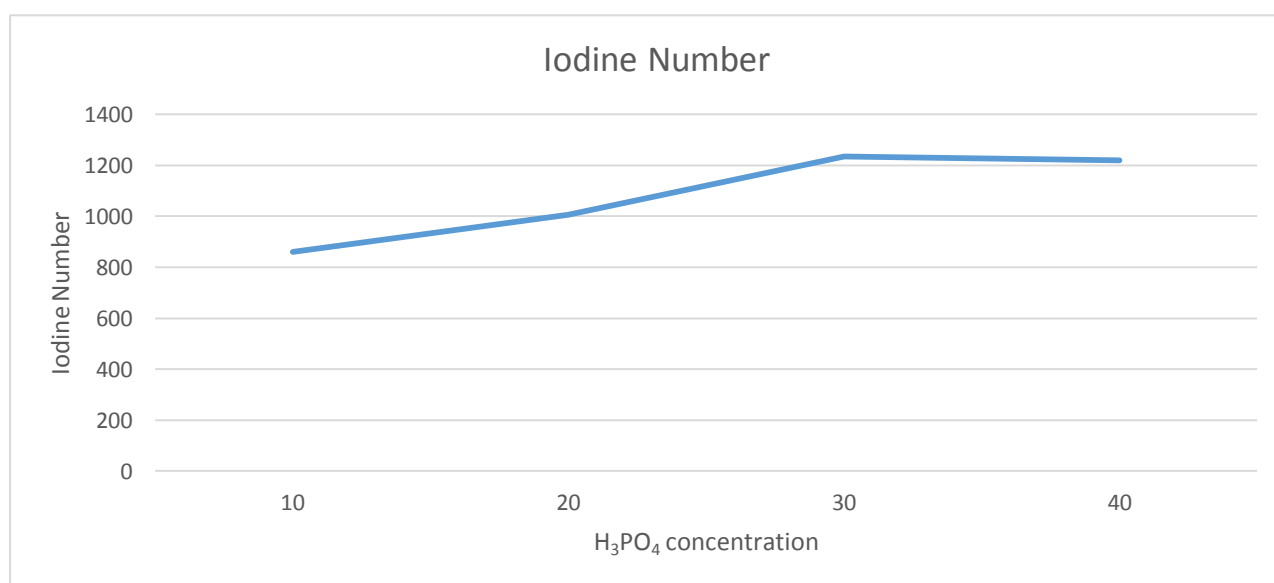


Figure 4.7 (a) : Variation of Iodine number with H_3PO_4 concentration

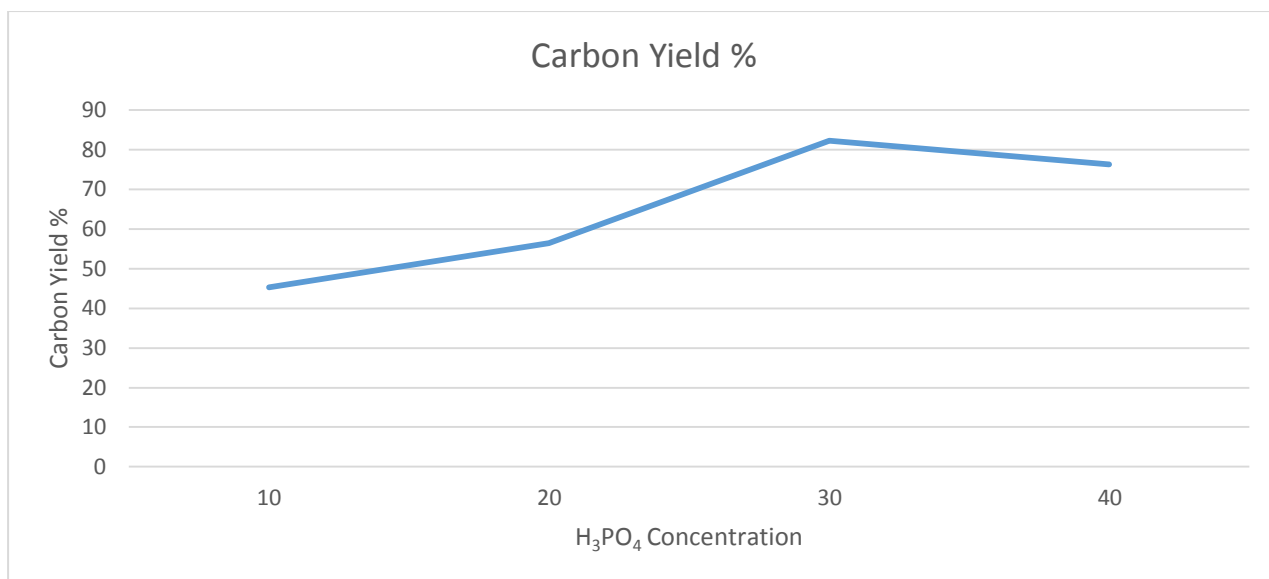


Figure 4.7 (b) : Variation of carbon yield with H₃PO₄ concentration

It is seen here that the Iodine number of the samples increases with the increase in concentration H₃PO₄ used and beyond 30% H₃PO₄ concentration there is a steady decline in the value of Iodine number since all the pores formed are either filled up or merge to form larger mesopores resulting in the decrease in iodine number as it is an indicator of micropores only. Similarly for Carbon Yield there is steady increase in the value and beyond 30% concentration, there is a steady decline in the yield as all the volatile matter is lost and the carbon macromolecules often react with the activating agent as a result of which CO₂ and other carbon compounds formed escape thereby reducing the carbon yield.

Table 4.5 : Iodine numbers and carbon yield of KOH impregnated samples

Temperatures	Impregnation Ratios					
	1:1		2:1		1:2	
	Iodine No	Yield(%)	Iodine No	Yield(%)	Iodine No.	Yield(%)
250°C	418	80.25	622	79	541	81.5
300°C	452	78	677	77	572	80

350°C	483	77.6	691	76	604	78
400°C	538	74.7	704	72	615	75
450°C	552	71.5	745	70	(618)	(73)
500°C	607	64	(748)	(61)	(619)	(65)

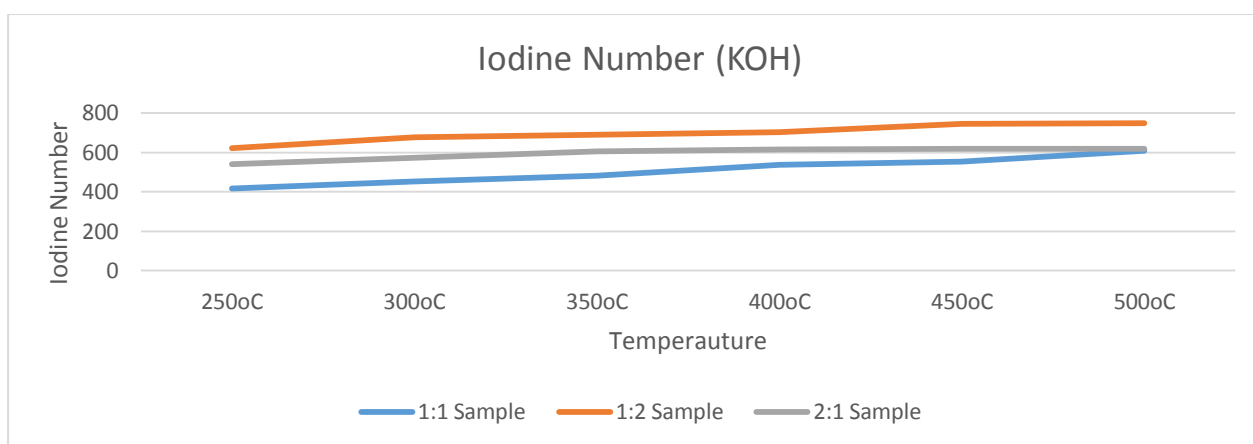


Figure 4.8 (a) : Variation of Iodine number with temperature for different KOH samples.

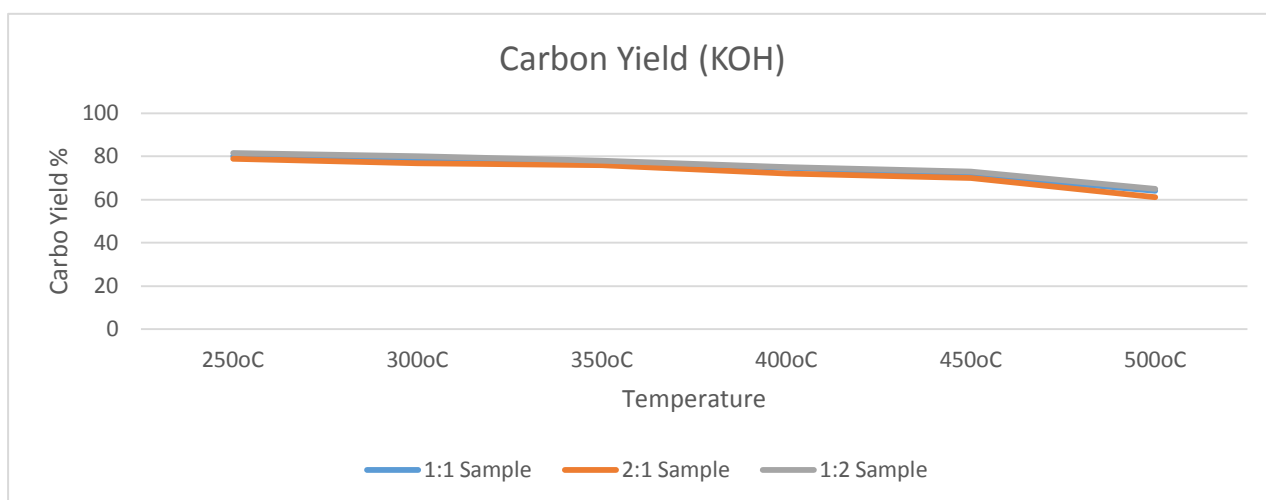


Figure 4.8 (b): Variation of Yield with temperature for different KOH samples

The different KOH impregnated samples (1:1, 2:1, 1:2) show an increase in Iodine Number values from 250°C to 500°C at which they get stabilized due to the pores getting completely occupied. Beyond this there is a decrease in Iodine number values as the smaller micropores merge to form large mesopores resulting in a decrease in iodine number which is an indicator of micropores. With decrease in micropores, iodine number thus also decreases. Similarly the carbon yield percentage decreases with an increase in temperature for all three samples as the volatile components are further lost and the macromolecules further react with the activating agent resulting in the loss of carbon in form of CO₂ and other compound which easily escape the surface of the molecule.

Table 4.6 : Iodine numbers and carbon yield of ZnCl₂ impregnated samples

Temperatures	Impregnation Ratios					
	1:1		2:1		1:2	
	Iodine No	Yield(%)	Iodine No	Yield(%)	Iodine No.	Yield(%)
400°C	(615)	(77)	(548)	(71)	741	82
450°C	(684)	(74)	(631)	(69)	892	79
500°C	752	72	686	68	961	74
550°C	824	66	755	64	1098	70
600°C	1166	64	782	60.5	1105	69
650°C	1173	58	782	56	(1112)	(67)

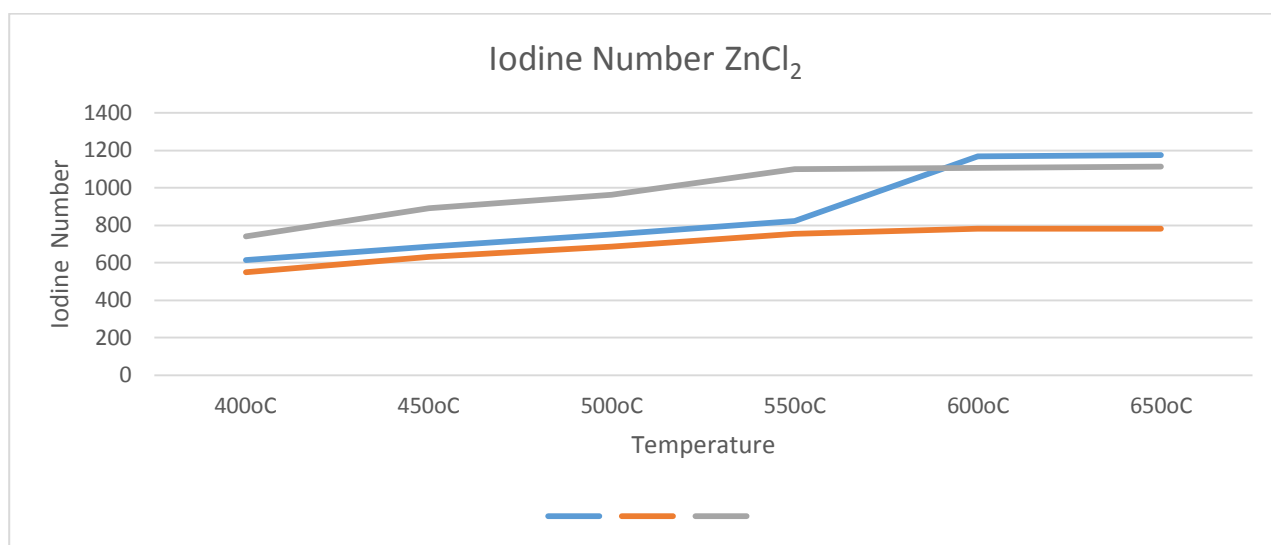


Figure 4.9 (a) : Variation of Iodine number with temperature for different ZnCl_2 samples

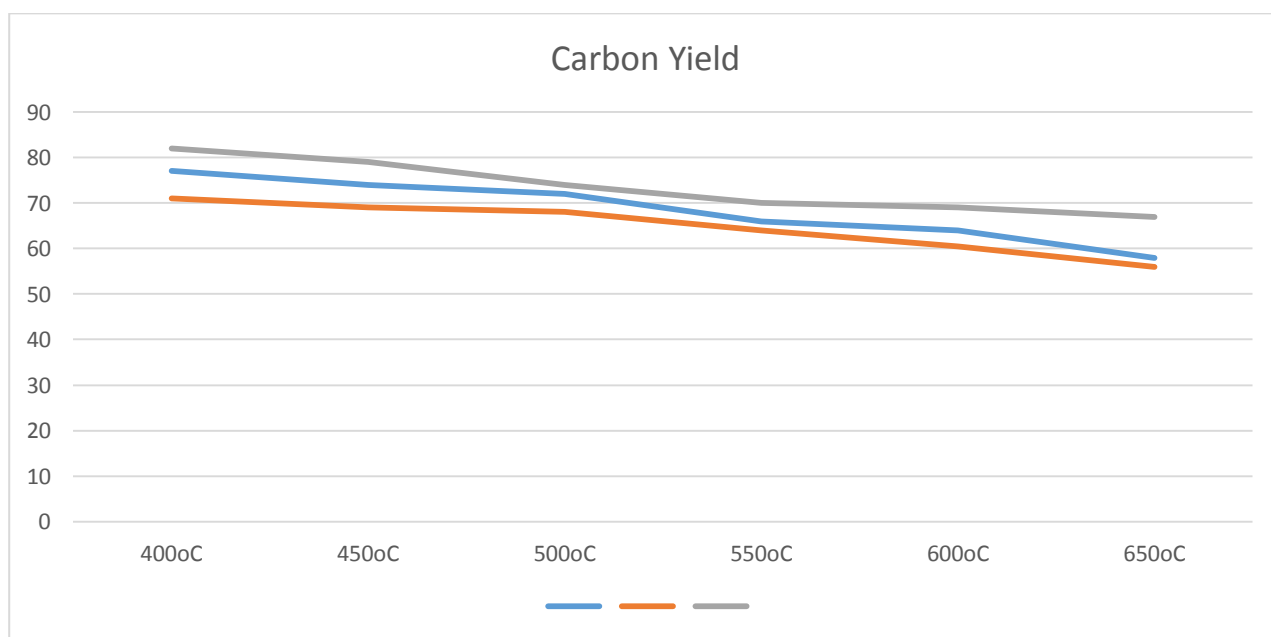


Figure 4.9 (b) : Variation of Yield with temperature for different ZnCl_2 samples

Iodine number shows an increase with temperature till the temperature range of 600°C-650°C beyond which it stabilizes owing to the complete occupation of the pores thus preventing further adsorption. Also the merging of smaller pores to form larger mesopores at higher temperatures affects the iodine number and carbon Yield. Carbon yield shows a steady

decline in value for all three samples (1:1, 2:1, 1:2) as the volatile component content keeps decreasing with increase in temperature.

Samples which exhibited the highest Iodine Numbers were further exposed to varying time and activation temperature and their yield and Iodine number were evaluated as shown in the table below. Samples which exhibited the highest Iodine number with a retention time of 30 minutes were then carbonized for a period of 60 minutes. The samples were also further carbonized at temperatures 50°C higher than the samples which showed the highest Iodine number.

Table 4.7 : Effect of carbonization temperature and time on iodine number and yield

Sample	Temperature	Time(in minutes)	Iodine Number	Yield(%)
H ₃ PO ₄	700°C	60	1221	77
H ₃ PO ₄	750°C	30	1193	72
KOH	450°C	60	752	67
ZnCl ₂	650°C	60	1172	62
ZnCl ₂	700°C	30	1185	57

H₃PO₄ impregnated activated carbon showed a highest iodine number of 1235 and a carbon yield of 82.3 % at a temperature of 700°C for a duration of 30 minutes. This sample was further carbonized at the same temperature for a duration of 60 minutes, which resulted in a slight decrease in iodine number which is a result of increase in thermal stress due to longer exposure to high temperatures which leads to enlarging and merging of the pores already formed thereby resulting in a decrease in micropores and thereby a decrease in iodine number. Also there was a decrease in yield owing to the escape of volatile matter which resulted in lesser yield.

The H_3PO_4 sample was also carbonized at 750°C for a duration of 30 minutes to check the temperature effects on iodine number and yield. The resulting iodine number and yield were lesser as compared to the sample at 700°C and a duration of 30 minutes, because as temperatures increase the formation of micropores decreases resulting in a decrease in iodine number and the yield also decreases due to the loss of volatile matter at higher temperature or on exposure to heat for a longer duration.

Similar is the case with KOH impregnated sample carbonized sample at 450°C for a duration of 60 minutes instead of 30 minutes and the ZnCl_2 impregnated samples in the table above. The KOH sample shows a marginal increase in iodine number which is only expected to further stabilize and decrease at higher temperatures.

The above table shows that there exists optimum temperature and duration required for the activated carbon to show good values of iodine number and carbon yield. Beyond these conditions, owing to the thermal effects and the presence of activating agent which often react with carbon components of the precursor, thereby resulting in a decrease of carbon yield, there is always a decrease in iodine number and carbon yield. Thus it is important to maintain optimum conditions for the desired quality of activated carbon.

4.1.5 OTHER CHARACTERISTICS

Other characteristics properties of the activated carbon samples prepared from the different impregnating agents were found by performing the standard tests for each of the characteristics.

Table 4.8 : Characteristics properties of activated carbon obtained at optimum conditions

Characteristic Properties	JPAC with KOH	JPAC with ZnCl_2	JPAC with H_3PO_4
pH	7.14	6.87	6.8
Carbon Yield	70 %	58 %	82.3

Moisture Content	2.22 %	2.6 %	4.2 %
Ash Content	14%	12 %	6.2 %
Volatile Matter	13.78 %	27.94 %	17.3
Density	1.25 g/cm ³	1.08 g/cm ³	0.72 g/cm ³
Specific Gravity	1.25	1.08	0.72
Porosity	0.76	0.82	0.81
Iodine number	745	1173	1235
Specific Surface Area	480 m ² /g	546 m ² /g	731 m ² /g

H₃PO₄ sample showed the highest Iodine number value at 700°C for a time period of 30 minutes (1235, yield-82.3 %). The samples were then subjected to carbonization at 700°C for a period of 60 minutes which showed an Iodine number of 1221 and a yield of 77 %. Whereas the sample carbonized at 750°C for a time period of 30 minutes showed an iodine number of 1193 and a yield of 72 %. This shows that a temperature of 700°C and a retention time of 30 minutes were the ideal conditions for preparation of activated carbon above which the iodine number and carbon yield show a decline.

The highest iodine number and yield among the KOH samples was shown by the 2:1 sample carbonized at 450°C (745-Iodine Number, yield-70 %), which was then carbonized at the same temperature for a time period of 60 minutes. This sample showed only a marginal increase in the iodine number value (752) but a decrease in the carbon yield percentage (67 %), which shows that the iodine number value was stabilizing and beyond this a decrease in iodine number values is to be expected.

The samples were thoroughly washed to bring the pH to around the 7 mark while the moisture content of all the three samples was found to be well below the 5 % mark.

The highest iodine number value among the ZnCl₂ samples was shown by the 1:1 sample carbonized at 650°C for a time period of 30 minutes (1173-iodine number, 58 %-yield) which was further carbonized at 650°C for 60 minutes and 700°C for 30 minutes, both of which resulted in

almost similar iodine number values (1172 and 1185 respectively) and almost similar or slightly lower yields indicating stabilization of the pores, further increase in temperature and time is only expected to further decrease the iodine number value and yield.

Other characteristics, such as pH, porosity, moisture content, ash and volatile matter content, of the samples which showed the highest iodine number value and yield were found out performing the standard tests for the respective characteristics.

The ash content of the H_3PO_4 sample was found to be quite low at 6.2 % whereas the KOH and ZnCl_2 samples showed a slightly higher ash content of 12 and 14 percentage respectively.

The volatile matter content of the KOH and H_3PO_4 samples were found to be below 20% while the ZnCl_2 sample showed a high volatile matter content of almost 28%.

The specific gravity of the three samples i.e H_3PO_4 , KOH, ZnCl_2 was found to be 0.72, 1.25, and 1.08 respectively whereas the porosity ratio of the samples was found to be optimum at 0.81, 0.76, and 0.82 respectively for H_3PO_4 , KOH, and ZnCl_2 respectively.

5. CONCLUSION

Powdered Activated carbon was prepared using jackfruit peel as the precursor. It was activated at temperatures based on TG analysis. Phosphoric Acid, Potassium Hydroxide, and Zinc Chloride were used as the activating agents in the preparation of Jackfruit Peel Activated Carbon. The precursor (Jackfruit Peel) was impregnated with the activating agents in different ratios (1:1, 2:1, 1:2) and (10%, 20%, 30%, 40%) for H_3PO_4 and subjected to activation. After activation using the activating agents, the samples were subjected to carbonization and their carbon yields were also calculated during the carbonization process. The samples were then subjected to the Iodine number test using the standard Iodine number test. The other characteristics of the samples which showed the highest Iodine number were then calculated their respective standard tests.

- The highest Iodine Number value of 1235 was observed for H_3PO_4 impregnated sample carbonized at 700°C for a duration of 30 minutes.
- The maximum Carbon Yield of 82.3 % was observed for H_3PO_4 impregnated sample carbonized at 700°C for a duration of 30 minutes.
- Among the different KOH impregnated and ZnCl_2 impregnated samples prepared, the ZnCl_2 sample prepared at a temperature of 650°C for a duration of 30 minutes showed an iodine number of 1173 and a carbon yield of 58 %.
- Compared to commercially prepared Activated Carbon which has an Iodine Number of 942, the JPAC was much higher-1235 in case of H_3PO_4 impregnated sample and 1173 in case of ZnCl_2 impregnated sample.

6. FUTURE SCOPE OF WORK

- Jackfruit peel activation could be performed using different retention times and temperatures and their properties studied, therefore by further optimization of the product, better quality activated carbon could be prepared.
- The properties of the prepared activated carbon could be compared with those of activated carbons prepared from other source materials, which could help in creating a better product.
- Adsorption studies of specific pollutants can be carried out using the prepared sample of activated carbon.

REFERENCES

- [1] A. Dabrowski, P. Podkoscielny, H. Hubicki and M. Barczak, "Adsorption of phenolic compounds by activated carbon," *Chemosphere*, 58, 1049-1070, 2004.
- [2] Z. Hongxia, W. Xiaoyun, L. Honghong, T. Tianshe and W. Wangsuo, "Adsorption behavior of Th(IV) onto illite: Effect of contact time, pH value, ionic strength, humic acid and temperature," *Applied Clay Science*, 127, 35-43, 2016.
- [3] B. N. Bhadra, P. W. Seo and S. H. Jung, "Adsorption of diclofenac sodium from water using oxidized activated carbon," *Chemical Engineering Journal*, 301, 27-33, 2016.
- [4] J. Sahira, A. Mandira, P. Bhadra Prasad and P. Raja Ram, "Effects of Activating Agents on the Activated Carbons Prepared from Lapi Seed Stone," *Research Journal of Chemical Sciences*, 3, 19-24, 2013.
- [5] R. T. Macia, M. J. Martinez and M. Hazelger, "Porous texture of activated carbon modified with carbohydrates," *Carbon*, 13, 30-36, 1997.
- [6] A. Buekens and A. Zyzykina, "Adsorbents and adsorption process for pollution control," *Pollution Control Technologies*, 4-11, 1990.
- [7] A. Bhatnagar and A. Minocha, "Conventional and non conventional adsorbents for removal of pollutants from water," *Indian Journal of Chemical Technology*, 13, 203-217, March 2006.
- [8] J. Menendez-Diaz and I. Martin-Gullon, "Types of carbon adsorbents and their production," *Activated Carbon Surfaces in Environmental Remediation*, vol. 12, pp. 1-46, March 2002.
- [9] S. Abechi, G. C.E, A. Uzairu and Y. Dallatu, "Preparation and Characterization of Activated Carbon from Palm Kernel Shell by Chemical Activation," *Research Journal of Chemical Sciences*, 14, 54-61, 2013.
- [10] B. Hameed, R. Krishni and Sata.S.A, "A novel agricultural waste adsorbent for the removal of cationic dye from aqueous solution," *Journal of Hazardous Materials*, 162, 9-17, 2009.
- [11] N. Bagheri and J. Abedi, "Preparation of high surface area activated carbon from corn by chemical activation using potassium hydroxide," *Chemical Engineering Research and Design*, 12, 1059-1064, 2009.
- [12] J. Raffiea Baseri, P. Palanisamy and P. Sivakumar, "Preparation and characterization of activated carbon from Thevetia Peruviana for the removal of dyes from textile waste water," *Advances in Applied Science Research*, 3, 377-383, 2012.

- [13] A. Verla, Horsfall.M, E. Verla, A. Spiff and O. Ekpete, "Preparation and characterization of activated carbon from fluted pumpkin seed shell," *Asian Journal of Natural and Applied Sciences*,1,39-51,2012.
- [14] R. Gottipati and S. Mishra, "Preparation of microporous activated carbon from Aegle marmelos fruit shell by KOH activation," *The Canadian Journal of Chemical Engineering*,91,1215-1223,2016.
- [15] B. Ash, P. Mukherjee, B. Nanda, J. Gumaste and B. Mishra, "Characterization and application of activated carbon prepared from waste coir pith," *Journal of Scientific and Industrial Research*,65,1008-1012,2006.
- [16] S. Yakout and G. Sharaf-El-Deen, "Characterization of activated carbon prepared by phosphoric acid activation of olive stones," *Arabian Journal of Chemistry*,14,1-8,2011.
- [17] I. Tan, A. Ahmad and B. Hameed, "Preparation of activated carbon from coconut husk: Optimization study on removal of 2,4,6-trichlorophenol using response surface methodology," *Journal of Hazardous Materials*,153,709-717,2009.
- [18] L. Jing, L. Jie, L. Yang-qing, S. Hai-sheng, Z. Zhi-an and L. Ye-xiang, "Influence of KOH activation techniques on pore structure and electrochemical property of carbon electrode materials," *Journal of Central South University of Technology*,13,360-366, 2006.
- [19] Sodeinde. O. A., "Preparation of a locally produced activated carbon from coconut shells and its use in reducing Hexammine Cobalt (III)," *International Journal of Chemical Engineering and Applications*,3,66-71,2012.
- [20] Y. Juan and Q. Ke-Qiang, "Preparation of Activated Carbon by chemical activation under vacuum," *Journal of Environmental Science and Technology*,43,3385-3390, 2009.
- [21] C.A. Oliveira, E. Pereira, I. R. Guimaraes, A. Vallone, M. Pereira, J. P. Mesquita and K. Sapag, "Preparation of activated carbons from coffee husks utilizing FeCl₃ and ZnCl₂ as activating agents," *Journal of Hazardous Materials*,165,87-94,2008.
- [22] K. Le Van and T. Thuy Luong Thi, "Activated carbon derived from rice husk by NaOH activation and its application in supercapacitor," *Progress in Natural Science:Materials International*,24,191-198,2014.
- [23] Baccara.R and F. Bouzida.J, "Preparation of activated carbon from Tunisian olive-waste cakes and its application for adsorption of heavy metal ions," *Journal of Hazardous Materials*,162,1522-1529,2008.
- [24] A. Halim Abdullah, A. Kassim, Z. Zainal, M. Zobir Hussien, D. Kuang, F. Ahmad and O. Sim Wooi, "Preparation and Characterization of Activated Carbon from Gelam Wood Bark," *Malaysian Journal of Analytical Sciences*,7,65-68,2000.

- [25] F. Liu, Z. Guo, H. Ling, Z. Huang and D. Tang, “Effect of pore structure on the adsorption of aqueous dyes to ordered mesoporous carbons,” *Microporous and Mesoporous Materials*, 227, 104-111, 2016.